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AMERICAN
DYESTUFF REPORTER

Devoted to Textile Chemistry
BLEACHING • DYEING • PRINTING • FINISHING

Contents

VOLUME 43 MARCH 29, 1954 NUMBER 7

• ***Special Article***

Fabric Surface Study 189
Ray Krammes and Charles Maresh

• ***Departments***

Book Reviews 192
Patent Digest 213
Paul Wengraf
Abstracts 216
Trade Notes and Calendar 220

• ***Proceedings of the American Association of Textile Chemists and Colorists***

AATCC Calendar P193
Employment Register P193
Reports from Local Sections P194
South Central and Northern New England Sections:—
Resistivity: One Clue to the Electrostatic Behavior of Fabric P195
Newton A Teixeira and Sidney M Edelstein
Rhode Island Section:—
New Neutral Dyeing Metalized Wool Colors .. P200
H F Clapham
1953 Annual Convention—Cotton Group:—
Practical Partial Acetylation of Cotton P203
Edmund M Buras, Jr, Albert S Cooper, Esmond J Keating and Charles F Goldthwait
New York Section:—
Dyeing and Finishing Piece Goods Made from Dynel-Rayon and Dynel-Rayon-Acetate Blends P209
F C Rippner
Membership Applications P212

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Contents of previous issues can be found by consulting the Industrial Arts Index and the Engineering Index, Inc, at your library

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*when treated with the polyantherenes or finished with urea of formaldehyde

AMERICAN
DYESTUFF REPORTER

VOLUME 43 MARCH 29, 1954 NUMBER 7

FABRIC SURFACE STUDY

RAY KRAMMES and CHARLES MARESH
Microscopical Laboratory, Calco Chemical Division
American Cyanamid Company, Bound Brook, N J

INTRODUCTION

IN this laboratory, we have adapted a replication technique for the study of glazed chintz fabrics. As is well known, the glazing of chintz and the embossing of fabrics is an old and much-used method of applying a desirable finish to fabrics. Until a few years ago, glazing effects were produced entirely by the use of nonpermanent finishes, such as common starch, together with the usual calender-roll techniques. Today more permanent finishing materials, such as melamine-formaldehyde, urea-formaldehyde, and others, are increasingly being employed.

It is often desirable to study microscopically the surface of a fabric after treatment with a finishing agent. This is done by the replication of the surface of the fabric and the preparation of optical photomicrographs, which provide a convenient method for comparison and record purposes.

The various techniques of surface replication have been employed increasingly during the past 15 years. Most of this work has been used in conjunction with electron microscopy where it is often found to be the only way to examine the surface of relatively large and opaque substances, such as metals *(1-9)*, fibers *(10-18)*, biological specimens *(19)*, and resin samples *(20)*. As long ago as 1891, J B Haycraft suggested the use of transparent replicas in the *Proceedings of the Royal Society*. The preparation of replicas for use in optical microscopy, however, has not become a common practice. Often, to many microscopists, the extra work involved does not appear to be justified. There are instances, however, where a replica method is the only way of yielding a satisfactory solution to a problem.

PROCEDURE

The replication of the glazed surface of the fabric with a thick (approx 10%) viscous water solution of methyl cellulose gives the basis for a true picture of the fabric surface. The methyl cellulose solution is placed on the fabric surface so as to cover an area approximately 2 cm in diameter. The water is removed by means of warm air which is blown over the fabric. By bending the cloth, the replica may be lifted away from it. This replica is an exact negative of the surface of the fabric as it exists without stress or strain.

The replica coated with aluminum metal at an angle serves to emphasize the differences between valleys and hills (warp or filling) of the fabric. The unit used for the evaporation of the aluminum metal is similar in general characteristics to other commercially available units, such as those sold by Radio Corporation of America, Optical Film Engineering Co, and Distillation Products, Inc. The unit consists of a good vacuum system inside of which there is place for the replica specimens and also a container for holding the metal prior to its melting and evaporation.

Figure 1 shows a schematic drawing of the evaporation chamber. In it is the tungsten wire basket supported at both ends by the posts through which the electrical current is applied. The aluminum metal, in the form of wire, is placed lengthwise inside the tungsten coil. The replica specimen is shown at an angle of 20°. Under vacuum conditions, metals evaporate with the particles traveling in straight lines as typified by the arrowed lines. The 20° angle was chosen so as to aid in distinguishing warp from filling threads. Approximately 0.05 gram of aluminum metal wire is used to give a satisfactory thickness of coating on the replica surface and the replica is placed 10 cm away from the aluminum wire. The hills of the replica receive a coating of the aluminum, whereas the valleys receive little or no metal. These aluminum-coated methyl cellulose replicas are then placed on a microscope slide, a drop of Shillaber's Immersion Oil is added and a cover glass is placed on the specimen. The slide may then be examined with an optical microscope set-up, and if desired, photomicrographs taken. Transmitted light is used for the examination; the areas having received more aluminum will transmit less light.

The photography of these replicas is of the low magnification type, the pictures being taken at a magnification of 50x. A microscope equipped with a 10x ocular, 4x objective, and a suitable bellows extention will give the desired magnification. The light source consists of a Bausch & Lomb housing with a ribbon filament lamp and the apparatus is adjusted for Kohler illumination. A medium-

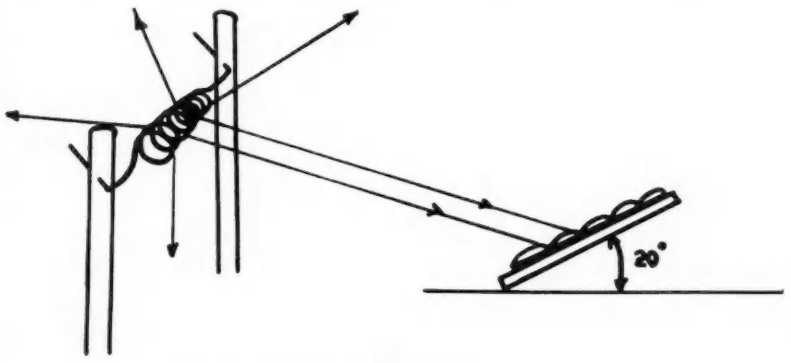
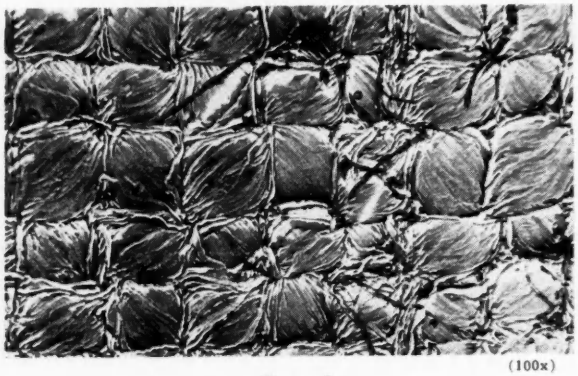


Figure 1
Schematic drawing of metals evaporation chamber.

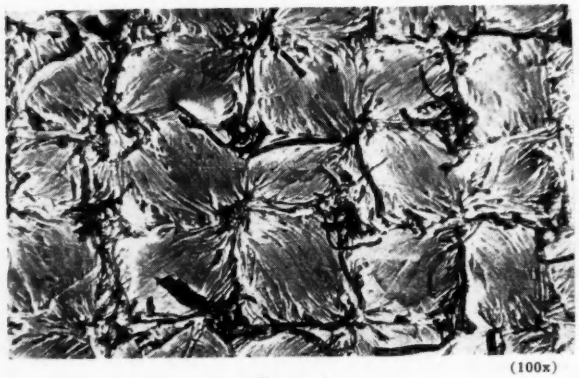
(100x)

Figure 2
Mercerized cotton, no resin treatment.



(100x)

Figure 3
Glazed cotton, no resin treatment.



(100x)

Figure 4
Cotton, melamine-formaldehyde-resin treated and glazed.

(100x)

Figure 5
Cotton, melamine - formaldehyde - resin treated, glazed and machine-washed.

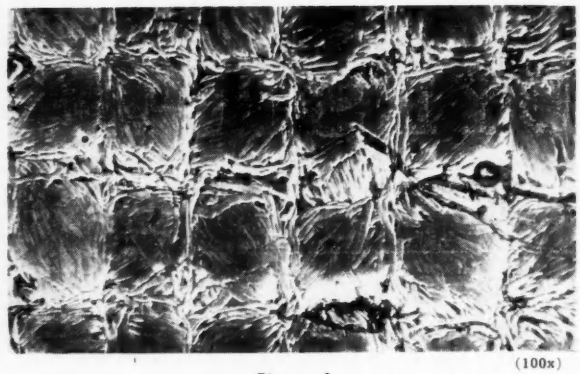


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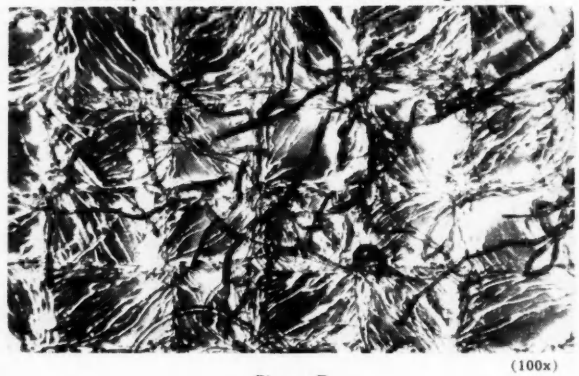
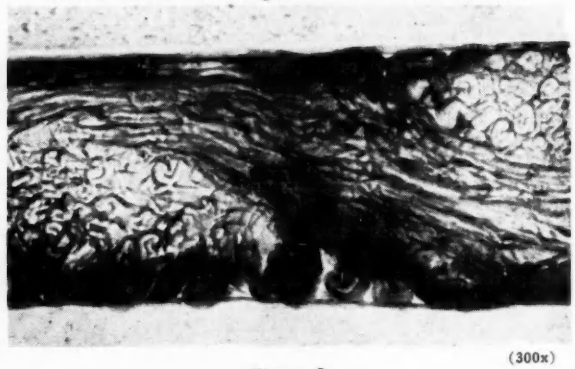
Figure 6
Cotton, a second finish which has been glazed.



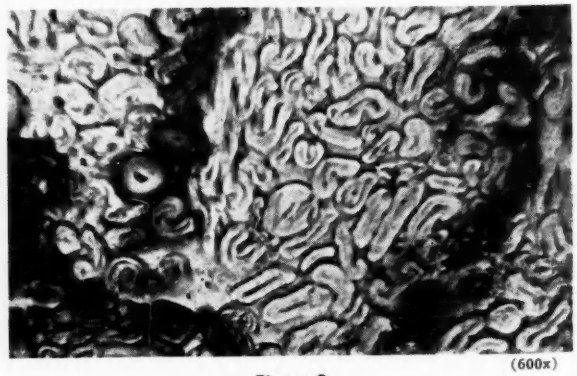
(100x)

Figure 7
Cotton, second finish, glazed and machine-washed.



(300x)

Figure 8
Resin-treated cotton cloth cross-section where staining of the resin shows it to be located essentially along the surface of the fabric.



(600x)

Figure 9
Cotton threads showing the location of the resin to be present in the darkly stained fibers.

speed film, such as Isopan (Agfa), XF Pan (Du Pont), or Super Ortho Press (Kodak), is satisfactory for the photomicrographs.

The photographs obtained by this procedure are not of the original fabric but of the replicas prepared from the fabric surface. By a second replication of the original replica, the fabric surface can be reproduced. This second replication procedure is usually not performed in this laboratory. In the single-replica method elevations in the original fabric become depressions, which do not receive any aluminum coating since the aluminum is applied at an angle of 20°. These depressions (elevation in the original) record in the finished prints as light areas or highlights and the depressions in the original fabric appear darker. Where the fabric is essentially in one plane with very few elevations and depressions a uniform deposit of aluminum is obtained and the desired effect of flatness is produced.

Figures 2-7 illustrate some of the photomicrographs obtained by this technique.

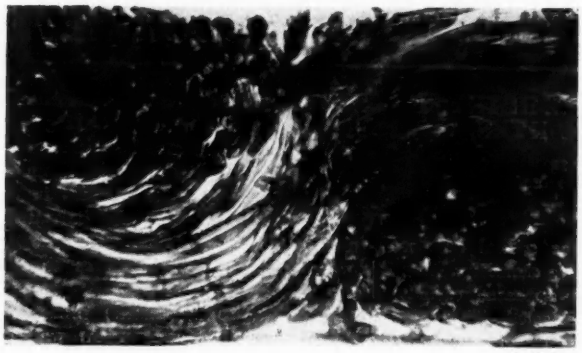
Figure 2 shows the surface of a piece of mercerized, but otherwise untreated, cotton cloth. The apparently smooth areas are actually deep places between the warp and filling threads so deep that they did not replicate.

Figure 3 illustrates another untreated cotton cloth which has been flattened or glazed by running over the glazing rolls. After laundering or normal usage this fabric rapidly approaches the condition shown in Figure 2.

The use of melamine-formaldehyde resin in addition to calender treatment results in a finish such as that shown in Figure 4, which is the original; Figure 5 shows a like piece of cloth after having received repeated machine washings. Note that little, if any, significant change has occurred in the physical characteristics of the glazed surface. The loose fibers present in most of the photomicrographs are probably caused by the adherence of fibers to the replica during stripping from the cloth.

Figures 6 and 7 are of a second available glaze finish before and after machine washing. Note that the machine washing has substantially changed the surface of this fabric indicating a less durable glazed finish.

Occasionally a glazed or embossed fabric, such as the untreated cotton, may be "wet" by the water in the methyl cellulose solution; if this happens an alternate method may be employed for obtaining the replica. A piece of the fabric is placed on a glass plate covered with a thin (0.03 mm) polystyrene film, which in turn is covered with a 1 x 3-inch microscope slide. A kilogram weight is placed on top of the glass-cloth-polystyrene-glass "sandwich", which is put into an oven at 110°C for 30 minutes. This "sandwich" is then allowed to cool to room temperature and the replica, when removed or stripped, is ready for aluminum coating as previously outlined for the methyl cellulose replica.



(300x)

Figure 10
Resin-treated cotton cloth where staining shows it to be completely penetrated.

A method for studying the location of the resin on the same glazed fabrics also makes use of microscopical techniques. This involves the staining of the resin, cross-sectioning, and again photographing. For this method to give the desired results, it is first necessary to use a dye that will color the resin and not the fabric, or inversely, to stain the fabric but not the resin. The melamine-formaldehyde-type finishes on cellulosic materials, such as cotton, are conveniently stained with Calcocid Alizarine Blue SAPG (C I 1054), a blue dye. This stain is prepared as a 0.1 percent water solution containing 1.0 percent of surfuric acid. The fabric is stained at the boil for five minutes and washed in water to remove excess stain. After drying, the sample as a fabric is cross-sectioned; wherever the resin is present the fibers are stained blue.

Figure 8 shows the cross-section of a resin-glazed fabric where essentially all of the resin is present near the glazed surface. The cotton that has received no resin is colorless.

Figure 9 illustrates an interesting result where the resin has been absorbed at the cotton-thread edges so that the inner fibers of the thread are devoid of resin. Figure 10 shows the fabric completely penetrated by the resin.

CONCLUSION

By the effective use of replication, metallic evaporation, and photomicrography, a true picture may be obtained of many types of fabric surfaces. These data are useful in the comparison of existing products and in the development of new finishes.

ACKNOWLEDGMENTS

Gratitude is expressed to G L Royer for his encouragement to publish this phase of our microscopical work.

Appreciation is also in order to the Textile Resin Department of American Cyanamid Company at Bound Brook, New Jersey for supplying the sample fabrics; of special mention in this department are T F Cooke, P Roth and G Moretti.

Thanks are also extended to P Buhl who did some of the laboratory and photographic work.

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BOOK REVIEWS

THE CHEMISTRY OF SYNTHETIC DYES, Volume II

K Venkataraman
Academic Press Inc
New York, N Y
$15.00; 1952

This is the second of a two-volume treatist on dye chemistry. The first volume, which appeared earlier, has been previously reviewed. The same high standards and comprehensivesness are maintained throughout the second volume.

The work consists of twenty chapters, an author index, a subject index, and a most valuable index of dyestuffs. A brief discussion of the various aspects of the text would seem in order.

The first seventeen chapters are concerned with dyestuffs not treated in Volume I. These are classified according to chemical structure and application properties. It would not seem advisable to catalogue the complete listing here. Certain especially noteworthy chapters, such as Chapter XXVII on Anthraquinone Dyes for Cellulose Acetate; Chapters XXX, XXXI, and XXXII on Anthraquinone Vats; Chapter XXXIII on Indigoid and Thioindigoid Dyes; Chapter XXXVII on Phthalocyanines, and Chapter XXXVIII on Cyanines, should be mentioned in light of modern interest, particularly in view of problems concerning dyeing the newer synthetics.

As in the first volume, each chapter on the various dyes is quite complete, well documented, and adequately illustrated with structural formulas.

A particularly valuable chapter on the action of light on dyes and fibers is included. A large amount of material previously scattered throughout various journals and in several languages is collected and correlated in this chapter. This fact alone warrants the purchase of the book by those concerned with such problems.

The chapter on constitution versus substantivity appears to be slightly brief, but this is probably in light of similar material covered by Dr Vickerstaff in his book concerning dyestuff characteristics. This should not be considered as an adverse comment, for the chapter is clearly written and quite constructive.

The last chapter deals with identification, analysis and evaluation of dyestuffs. It was a pleasant surprise to the reviewer to find the valuable technique of chromatography treated in considerable detail. This has not been the case of previous texts on dyestuffs. The scheme of analysis is the familiar and well-tried Green-Clayton method.

The discussion of colorimetric methods is somewhat disappointing in that it is so short and also that it appears to reflect a viewpoint differing from the opinion of many color workers in this country. However, it should be stated that there is considerable difference of opinion among individuals as to the merit of various photoelectric comparative (or spectrophotometric) procedures, and another reviewer might well consider the treatment perfectly adequate.

In reviewing the first volume the fact that certain standard texts were not referenced was commented on. It should be mentioned that references to these texts appear in the second volume. However, it would seem that some sort of cross reference would be in order, although this difficulty may have arisen due to the volumes appearing at separate times.

The structual problems of cellulose are discussed in considerable detail in this volume and perhaps the comments made along these lines in the first review should be somewhat tempered.

Although a few criticisms have been made in both the first review and here, it should be made clear that these are relatively minor, to be considered possibly in future revisions. These two volumes comprise the most exhaustive and authorative treatise available today. Every person concerned with dyestuff chemistry, both from the research and application viewpoints, should have these volumes near at hand. The author is to be heartily congratulated for performing a task which can only be considered in proper perspective by others who have undertaken similar endeavors. Although the price of approximately $30.00 for the two volumes may seem high, one could readily use up thirty dollars worth of time looking for information which can be found in a few minutes in these books.—RED

ENCYCLOPEDIA OF CHEMICAL TECHNOLOGY, Volume 10—Pentacene to Polymethene Dyes

Edited by Raymond E Kirk and Donald F Othmer
The Interscience Encyclopedia Inc
New York 1, N. Y.
xvi+ 976 pp
Sub Price: $25.00; 1953

This book is part of a larger and monumental series of volumes on chemical technology, which is published at intervals of six months. Volume 10 covers subjects of interest ranging from pentacene to polymethene dyes. It is certainly an ambitious venture, and a successful one, so successful indeed that it is recommended that any technical library should have not only this but all the other published volumes.

Many topics of interest to the textile industry are covered: Organic and Inorganic Peroxides, Phenyldiamines, Phosphoric Acid and Phosphates, Photochemistry, Organic and Inorganic Pigments, Plant Layout and Location, Plastics, Polyamides, Polyethylene, and Polymethene Dyes. Each is covered in a comprehensive fashion concerning materials, methods, processes, and equipment. In addition, at the end of each article a bibliography section is included.—VL

CONDENSED PYRIDAZINE AND PYRAZINE RINGS (CINNOLINES, PHTHALAZINES, AND QUINOXALINES)

J C E Simpson
Interscience Publishers, Inc
250 Fifth Avenue
New York 1, N Y
$12.50 (or $11.25 subscription price); 1953

The chemistry of heterocyclic compounds is considered in this book with the subject matter divided into three sections: 1) cinnolines, 2) phthalazines, and 3) quinoxalines. Numerous tables, structural formulas and references are contained.

The subject matter, dealing with one of the most complex branches of organic chemistry, is covered extensively and as such would appeal to organic chemists, especially those interested in the chemistry of heterocyclic compounds.—HB

ORGANIC SYNTHESES, Volume 33

Charles C Price, Editor-in-chief
John Wiley & Sons, Inc
440 Fourth Avenue
New York 16, N Y
$3.50; Nov 23, 1953

As stated in the title, this book is an annual publication of satisfactory methods for the preparation of organic chemicals. The present volume describes the preparation of 40 compounds by means of tested procedures and also lists alternate methods of synthesis described in the literature. A subject index comprises material from Volumes 30 to 33. The book is a reference volume and is of primary interest to the synthetic organic chemist. —JHR

(Additional book reviews on page 219)

Proceedings of the

American Association of Textile Chemists and Colorists

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Editor of Proceedings: Carl Z Draves, 30 Lahey St, New Hyde Park, N Y

MARCH 29, 1954

AATCC CALENDAR

COUNCIL
1954—Apr 30 (Atlantic City), June 15, Sept 15, Nov 19.
1955—Jan 21, Apr 15, June 17

NATIONAL CONVENTIONS
Sept 15-18, 1954 (Atlanta Biltmore, Atlanta, Ga); Sept 21-23, 1955 (Chalfonte-Haddon Hall, Atlantic City, N J; 1956 (New York); 1957 (Boston).

HUDSON-MOHAWK SECTION
April 9, May 14 (Albany); June 18 (Outing).

MID-WEST SECTION
April 10 (Netherlands Plaza, Cincinnati, O); June 11-13 (Annual Outing—Lake Lawn Lodge, Delavan, Wis).

NEW YORK SECTION
April 9, May 14 (Swiss Chalet); June 18 (Outing, North Jersey Country Club, Wayne, N J).

NORTHERN NEW ENGLAND SECTION
Apr 9 (Lowell Tech, Lowell, Mass); June 11 (Outing—Merrimack Valley C C, Methuen, Mass); Oct 8 (Colonial C C, Lynnfield, Mass); Dec 10 (Woodland Golf Club, Auburndale, Mass).

PIEDMONT SECTION
Apr 10 (Hotel Roanoke, Roanoke, Va); June 11-12 (Outing, Mayview Manor, Blowing Rock, N C); Oct 16 (Hotel Barringer, Charlotte, N C).

PHILADELPHIA SECTION
Apr 9 (Kugler's Restaurant, Philadelphia, Pa); May 21 (Outing—Manufacturers Golf & Country Club, Oreland, Pa); Sept 24, Oct 29 (Penn-Sherwood Hotel, Philadelphia, Pa); Dec 3 (Kugler's).

RHODE ISLAND SECTION
Apr 29 (Wannamoisett Country Club, Rumford, R I); June 11 (Outing—Wannamoisett C C); Oct 14 (Johnson's); Dec 2 (Annual Meeting—Sheraton Biltmore, Providence, R I).

SOUTH CENTRAL SECTION
April 24 (Hotel Patten, Chattanooga, Tenn); June 4-5 (Summer Outing—Chattanooga Golf and Country Club); Dec 4 (Hotel Patten, Chattanooga).

SOUTHEASTERN SECTION
May 14-15 (Outing—Radium Springs, Ga); Dec 11 (Atlanta-Biltmore, Atlanta, Ga).

WASHINGTON SECTION
April 9 (National Institute of Dry Cleaning, Silver Spring, Md).

WESTERN NEW ENGLAND SECTION
May 14 (Ladies Night—Waverly Inn, Cheshire, Conn); June 18 (Outing—Chase Country Club, Waterbury, Conn); Oct 1, Nov 12, Dec 10 (Rapp's).

EMPLOYMENT REGISTER

This column is open for two insertions per year, per member, without charge. Blanks can be obtained from, and filed with, the Secretary of the Association, Lowell Textile Institute, Lowell, Mass. It is understood that these will be open to inspection by prospective employers who can obtain further information from the Secretary.

54-4

Education: B S, textile chemistry & dyeing.

Experience: Supervision in production of synthetic fibers.

Age: 31, married; veteran; references; South preferred.

3-29, 4-12

54-5

Education: Textile chemistry & dyeing.
Experience: Textile chemist & dyer.

Age: 37; married; veteran; references; northeastern US preferred.

3-29, 4-12

Membership Cards

Membership cards have now been mailed to all members who have paid their dues. Any member not receiving his card within two weeks of payment should notify the Secretary:

Dr H C Chapin, Secretary
American Association of
Textile Chemists and Colorists
Box 28, Lowell, Massachusetts

"THE APPLICATION OF VAT DYES"

AATCC Monograph No. 2

Price $5 per copy, postpaid, to members; to nonmembers it is $6 per copy. Send your check or money order to Dr H C Chapin, P O Box 28, Lowell, Mass.

"The group of fifteen editors responsible for this monograph consists of a well-balanced cross-section of American industry concerned with the production, marketing and application of vat dyes. The AATCC is to be congratulated on the production of a comprehensive survey of American vat dyeing and printing practice which is the major theme of this application . . . the book will be useful as a work of reference at this side of the Atlantic and as a working manual for the American colorists for whom it was designed. . . . If a clear account of American practice is required . . . the enquirer *must* have access to this volume".—*JOURNAL OF THE TEXTILE INSTITUTE* (England).

"This book . . . is abreast of the latest technical developments and research. Its value is increased because of the lack of books in this field today . . . we can readily recommend this book to all technical and practical men".—*MELLIAND TEXTILBERICHTE* (Germany).

"This monograph contains the work of many mill-experienced authors, plus good, sound chemical and application principles developed in the laboratories and mills enjoying AATCC membership. . . . This book puts between two covers much information that a dyer is constantly searching for. It is a good investment for any laboratory, dyehouse, or printshop."—*TEXTILE WORLD*.

". . . this monograph should be in the hands of every user of vat dyestuffs, every student and every teacher. The book is well indexed and includes valuable lists of equivalent trade names. It will certainly take its place as a standard book of reference".—*JOURNAL OF THE SOCIETY OF DYERS AND COLOURISTS* (England).

Save this date . . .

MAY 19, 1954

All-day Meeting • Hotel Statler, New York

. . . details later

REPORTS FROM THE LOCAL SECTIONS

Philadelphia Textile Institute Student Chapter

Top (l to r): Berman, Heppenstall, McDonald, Nuttall, Muscelli, Obidinski, Zavaglia, Fawcett
Center: Gropper, Heller, Snyder, Tetzner, Sugarman, Foster, Patel
Bottom: Rassie, Celikiz, Weiss (secretary), Larrat (chairman), Demuth (co-chairman), Nelson, Ayanoglu, Schrieber

THE Philadelphia Textile Institute Student Chapter met on January 14, 1954 with approximately 75 members and guests present to see the motion picture "Glass for Science." The color-sound film, shown through the courtesy of the Corning Glass Works, Corning, N Y, told the story of the manufacture of glassware for the chemical industries. A discussion of new products being made from glass followed.

Thirty members, including seven foreign students, now comprise the Chapter.

—♦ ♦—

Mid-West Section

THE Spring meeting of the Mid-West Section will be held April 10 at the Netherlands Plaza Hotel, Cincinnati, Ohio.

The afternoon session will feature a talk by Henry E Millson, assistant technical director, Application Research Department, Dyestuff Division, American Cyanamid Co, on "The Effect of Sequestering Agents on Cotton and Wool Dyes."

Jean Kern, Dye Application Laboratories, National Aniline Division, Allied Chemical & Dye Corp, will speak on "The Dyeability of Modern Synthetics" at the evening session.

—♦ ♦—

Western New England Section

THE Annual Outing of the Western New England Section will be held on June 18th at the Chase Country Club, Waterbury, Conn. John B Dick, The Pond Lily Co, is Chairman of the Outing Committee.

Hudson-Mohawk Section

THIRTY-FIVE members attended the Hudson-Mohawk Section's dinner and technical program on March 5, 1954 at Jack's Restaurant, Albany, N Y.

The speaker of the evening was David C Scott, president, Scott Testers, Inc, who described in detail many of the new developments in textile testing equipment and the reasons for such development.

—♦ ♦—

Washington Section

APPROXIMATELY 80 members and guests of the Washington Section met on March 5th at the Textile Museum, Washington, D C, to hear Miss Louisa Bellinger, the museum's curator-analyst, deliver a talk on "Textiles of the Ancients." Following a guided tour of the museum's exhibits, an informal discussion period was held.

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Philadelphia Section

THE Philadelphia Section held its second meeting of the year on February 26, 1954 at Kugler's Restaurant, Philadelphia, Pa, with approximately 136 members and guests in attendance.

Speaker of the evening was Raymond W Jacoby, Ciba Co, Inc, on the subject "Textile Dyeing, Printing and Finishing—American and European Plan." Following Mr Jacoby's talk, J A Woodruff, American Viscose Corp, presented a resume of the new developments concerning flammability in the textile industry and discussed the latest Federal Trade Commission rulings in that regard.

The next meeting of the Philadelphia Section will be held at Kugler's on April 9th at which time Jules Labarthe, senior fellow at the Mellon Institute of Industrial Research, will speak on "Ten Thousand and One Consumer Complaints." Dr Labarthe will discuss the progress attained in setting up standards for textile products bought by hotels, motels, hospitals, and other large institutional buyers. At the same meeting Jack A Adams, Parr Engineering Co, will discuss the work of the AATCC Committee on Stream Pollution.

—♦ ♦—

New York Section

A MEETING of the New York Section was held on Friday evening, February 26, 1954 at the Hotel Statler, New York. A dinner preceded the meeting.

Arnold Silver, superintendent of finishing at Brewster Finishing Co, presented a paper entitled "Resin Finishing of Fabrics" and R J Thomas, Technical Laboratory, E I du Pont de Nemours & Co, Inc, discussed "Absorption of Disperse Dyes by Dacron." Co-authors with Dr Thomas were J J Iannarone and Mrs O S Larson, also of DuPont's Technical Laboratory.

The attendance was approximately 300.

—♦ ♦—

NNE-LTI Joint Meeting

THE Northern New England Section will hold its annual joint meeting with the Lowell Technological Institute Student Chapter on April 9 at the Institute.

The afternoon program will consist of a series of demonstrations by students employing various instruments used in textile research, such as the electron microscope, the G E Recording Spectrophotometer, the spectrograph, and the Instron. Participants will include Leo Barish, Nassos Anninos, David Kwok and Thomas Garvey. Experiments in papermaking and converting and leather finishing will also be featured.

The evening program will include the following papers: "Acetate Dyeing: Interfacial Film Phenomena"—Edwin C Sherburne; "Friction and Lubrication of Nylon Parachute Materials"—Harry J Demas; and "Wool Dyeing: Effect of Dye and Acid Valence"—Joseph B Levy.

South Central and Northern New England Sections——

RESISTIVITY: ONE CLUE TO THE ELECTROSTATIC BEHAVIOR OF FABRIC*

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Fabric Research Laboratories, Inc
Boston, Mass

and

SIDNEY M EDELSTEIN
Technical Director
Dexter Chemical Corporation
New York, N Y

To help readers understand the problem of static electricity in textile processing, the authors explain the fundamental principles of this phenomenon, which has intrigued scientists much longer than current electricity. They begin with definitions, then explain how charges are developed, and describe the mechanisms by which charges of static electricity are dissipated. The measurements to which a textile may be subjected to measure its tendency to become electrified and the effect of antistatic agents and atmospheric conditions on these measurements are shown and discussed.

The conclusion emphasized by the authors is that practical mill experience with an antistatic agent on a particular fiber is more important for evaluation of effectiveness than measurements.

I INTRODUCTION

"STATIC" problems in textiles arise almost always because electric charges accumulate on some materials until dangerous or annoying charge densities are reached. These problems have been accentuated by the introduction of new textile fibers, which are particularly susceptible to charge accumulation. The undesirable effects of this "static" have created problems for both the manufacturers and users of textiles *(1)*.

Although the study of electrostatics (the behavior of fixed charges) has occupied many of the most brilliant minds in the history of science, the theoretical explanations for many common "static" phenomena remain problems for the atomic physicist *(2, 3)*. There is, however, a great body of work available on the experimental investigation and less fundamental theory of electrostatic behavior to guide the development of procedures for measuring and controlling the electrification of materials used in the textile industry.

Proper evaluation of the electrostatic nature of a fabric requires the measurement of all the various mechanisms for charge generation and charge dissipation. While satisfactory instruments for all of these measurements are not yet available, the measurement of one particular electrical property of a textile can be helpful in research and in obtaining information of a broad nature. The measurement of the specific resistance, or resistivity, of a fabric, while by no means the whole answer to the problem, does furnish an indication of an important property influencing its electrostatic behavior *(4, 5, 6, 7, 8, 9)*.

An improved instrument for measuring the resistivity of textiles under carefully controlled conditions has been built and already has been used to obtain practical information on the behavior of chemical antistatic agents. After a brief discussion of the physical principles involved, the theory of the design and construction of this instrument is presented.

* This report is based on part of a program being conducted at Fabric Research Laboratories, Inc, under the sponsorship of the Dexter Chemical Corporation. Presented by S M Edelstein before the South Central Section in Chattanooga, Tenn, on Dec 5, 1953, and before the Northern New England Section in Cambridge, Mass on Feb 12, 1954. Submitted to the Reporter by the Editor of Proceedings on January 26, 1954.

FUNDAMENTALS

DEFINITIONS —— In order to describe electrostatic phenomena, the following concepts are useful *(10)*:

The fundamental quantity of electricity is charge, and the electron, which is the smallest divisible element of electricity, is the intrinsic *unit of charge*. The work per unit distance required to separate two opposite charges is the electrical potential, expressed in volts. When two opposite charges that have been separated are allowed to come together, they liberate a certain amount of energy per unit distance; therefore, the use of the term *potential* indicates a potential energy per unit separation of two charges.

The *capacitance* of a system is a measure of the quantity of charge which can be kept separated by a given electrical potential or voltage. For the common parallel-plate capacitor, the capacitance depends inversely upon the separation and directly upon the area of the plates and the *dielectric constant* of the material between them.

The *dielectric constant* is the name for that property of a material which reduces the effect of the electrical field within that material. (This is *not* dielectric strength, which is the breakdown strength of an insulator.)

To pass a steady flow of electric charge, or electric current, through any material requires a voltage, and the ratio of the voltage to the current is called *electrical resistance*. The lower the resistance, the more easily a current passes through a material. Actually, however, the flow of charge may take place through the volume of the material or along its surface. Leakage current through any given material is composed of a *volume current* and a *surface current*, the latter depending very much on the condition and purity of the surface.

The earth, or *ground*, contains so many free charges that it acts as a very large reservoir, the depth of which (analogous to voltage) is not affected by the addition of even fairly large amounts of charge. Since everything on earth has a definite resistance and capacitance to ground, unbalanced electrical charges tend to return to ground.

The problem of excessive charge accumulation resolves itself into two components, which will be discussed separately: charge generation and charge dissipation, or charge de-electrification.

CHARGE GENERATION —— The mechanisms of charge generation in dielectrics, or nonconductors, are several, namely *(11)*:

A) *Electrolytic*, in which ions or charged particles are separated in solution and preferentially deposited on a nonconducting surface, where they remain as fixed charges.

B) *Double-Layer Separation*, or *Volta Mechanism*, in which a high, direct voltage is generated by contacting and then separating two different materials. This voltage is due to the *contact potential (12)*, which is a measure of the work per unit charge that must be performed to move a free electron (one not bound to any particular atom in the molecular structure) from one material to another when both are in contact.

C) *Spray Electrification*, in which charge is sprayed from a point source or series of point sources to a nonconductor by means of a high-voltage field.

D) *Frictional*, or *Triboelectric*, which is the least clearly defined mechanism but is usually considered to be the cause of that charge generated only by actual friction. As discussed later, some investi-

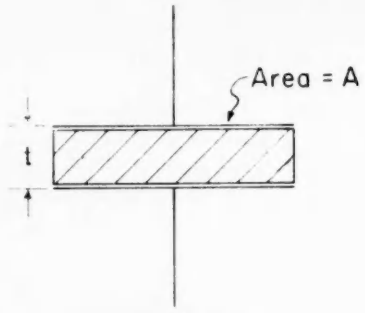

Figure 1
Volume Resistivity

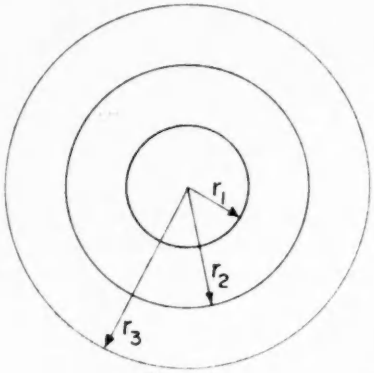

Figure 2
Surface Resistivity

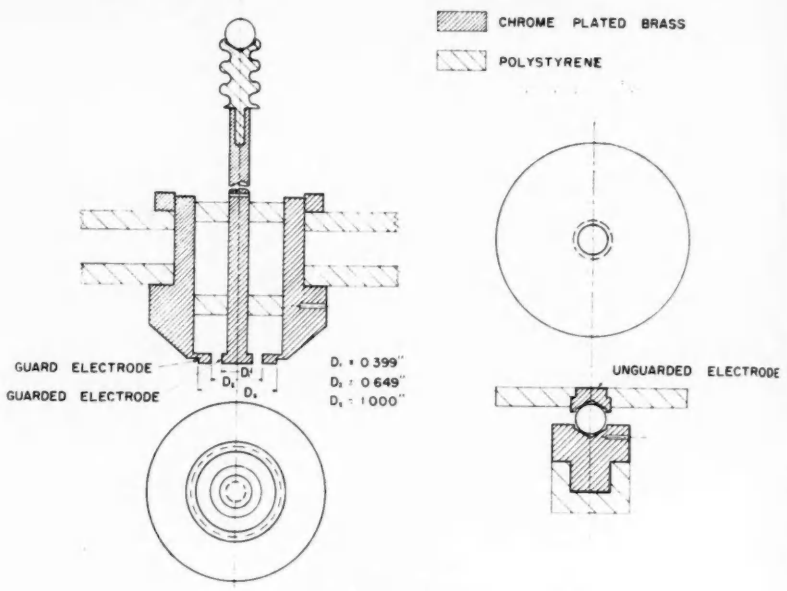

Figure 3a
Electrode for Measuring Resistance

gators believe the larger portion of this effect to be attributable to the double-layer mechanism.

E) *Mechanical Separation of Charges Created in Gases or Flames by Electrical Phenomena,* which accounts for the electrification (or de-electrification) of plastics held over a flame or against a swift stream of gas.

In the textile industry the most important mechanisms seem to be triboelectrification and double layer.

CHARGE DISSIPATION——There are several mechanisms of de-electrification which limit the ability of a textile to retain an excess of electric charge.

A) *Leakage to Ground,* by which the charge may move along the surface or through the volume of the material to be neutralized by the large number of free charges in the ground. Any comparatively large reservoir of free charges may act as a ground.

B) *Self-Neutralization Within Small Areas* by which it is also possible for charges of opposite polarity to neutralize each other by moving across the surface of a fiber or fabric. The surface of a charged insulator is not covered with a uniform layer of positive or negative charges but is a mosaic of small patches of positive or negative charge *(13).* These individual patches may be as small as a large molecule or big enough to be visible. Only the net effect of these charged areas is usually measured. It is possible, under conditions of limited surface conductivity, for a substantial diminution of static to occur by self-neutralization with no connection to ground.

C) *Corona-type Leakage,* another mechanism for the de-electrification of dielectrics by actual discharge into the atmosphere. If the density of charge is great, the electric field intensity, or the electric potential per unit distance, is also great, and when this intensity reaches a critical value, the air no longer acts as a dielectric. A type of glow discharge or corona occurs. (This type of discharge is often observed at the wing tips of airplanes, where it is called "St Elmo's fire.") Certain vapors, when mixed with air, increase this critical value of the electric field intensity, allowing a given system to attain a higher charge density. Carbon tetrachloride vapor increases the critical value; water vapor decreases this value, hence a partial explanation for the well-known fact that under conditions of high humidity less charge can be deposited on a given system *(14).* This effect is separate and distinct from the increase in conductivity of hygroscopic materials. Thus, any addition to a fabric which would lower the critical electric field intensity at the surface of a material would lower maximum charge density which could be maintained on that material.

D) *De-ionization,* also another effect attributable to the atmosphere. Unless air, or any other fluid, is especially purified, it will contain small particles of microscopical size which carry charges. There are also present ionized gas molecules, which, together with the small charged particles, act to de-electrify a dielectric by actual neutralization, since opposite charges attract and neutralize each other. Many systems for reducing electrostatic charge on dielectrics depend upon ionization of the air by sparks, radio-frequency fields, or radioactivity. Radioactive substances emit particles which can either ionize the air or directly neutralize the charge on the dielectric.

To summarize the discussion of generation and de-electrification, the generation is caused by the bringing together and separation of dissimilar materials *(15),* the combination having a relatively large contact potential. Whether or not actual rubbing is necessary is a question still being investigated by those conducting fundamental research *(16).* De-electrification can be accomplished by conduction through the volume or along the surface of the material, by neutralization with free ions and charged particles in the air, by glow-type discharge, or by direct neutralization through the action of radioactively emitted charged particles.

METHODS FOR MEASURING ELECTROSTATIC PROPERTIES ——— It should be possible to duplicate the charge situation responsible for the undesired effects and to measure the amount, distribution, and rate of decay of charge on a sample material.

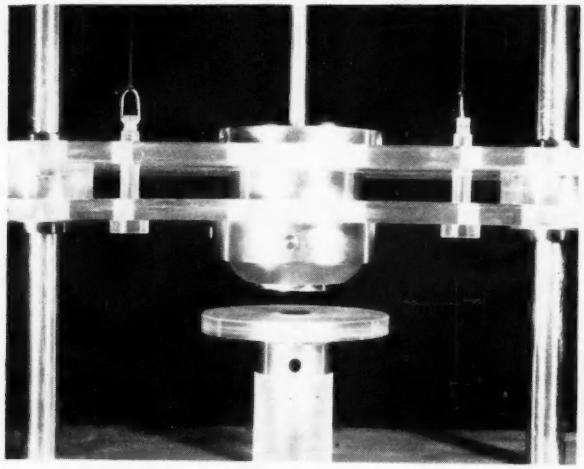

Figure 3b

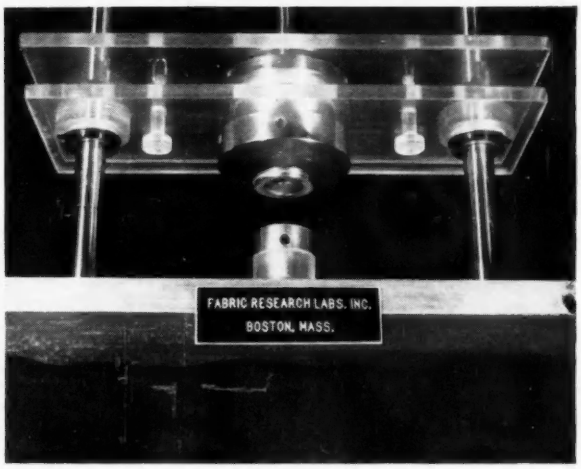

Figure 3c

Unfortunately, it seems to be even more difficult to deposit a uniform layer of charge on a nonconducting surface than it is to measure this charge. Most materials in a charged state have a motley pattern of positive and negative excess charge with varying charge densities. The regions of like charge are often too small to be located with even a relatively small searching electrode.

In addition to the nonuniformity of charge dispersion, there are three other reasons why charge is so difficult to measure. Since charge can only be measured by the current generated by its motion or the voltage generated by its accumulation, there must be at least twenty times the amount of available charge as is required to operate the measuring instrument. The three reasons for difficulty in measuring charge are the following:

a) There is a relatively small amount of charge per unit area on any insulator.

b) The resistance of most measuring instruments is too low compared with that between two points of the insulator.

c) The capacitance of most measuring instruments is too high compared with that between two points of the insulator where the charge is to be measured.

The difficulty in measuring electrostatic charges has resulted in more concentrated study of the dissipation of charges, which is more practical as well as more feasible. Theoretically, it is possible to match two surfaces so that no charge will be generated when the two surfaces are contacted and then separated *(17)*. Practically, however, the solution to the problem must be in preventing the accumulation and retention of whatever charge may be generated.

EXPERIMENTAL

MEASUREMENT OF RESISTIVITY ——Having presented some of the background to the general problem of electrostatic charges on textile materials, in the remainder of this discussion we shall deal with the measurement and use of the resistivity, or normalized resistance, of fabrics. The electrostatic fundamentals were reviewed mainly as a reminder that resistance, or resistivity, can at best furnish only a clue to one mechanism of charge dissipation.

In the simplest type of electrostatic problem the fabric acts as an insulator, that is, its resistivity is so high that it prevents the leakage of charge from some other body. This is the type of problem in which resistivity data can be most useful. Resistivity tests may also be used in quality control.

Before a discussion of the limitations of these tests in research, an instrument facilitating the measurement of resistivity will be described.

The resistance of a solid substance was defined above as the ratio between the voltage impressed across the material and the resultant current flow between the electrodes. In order to determine the inherent resistance, or *resistivity*, of a three-dimensional body, it is necessary to learn the paths taken by the current. Although the current may travel throughout the solid, there are two general paths it will take. The first, and most easily explainable, is through the volume of the material. This path has a certain electrical resistance, which is called *volume resistance*. Another path is along the surface of the material. This path has *surface resistance*. In a perfect vacuum, a perfectly clean or newly cut surface of the solid would be chemically and electrically the same as the inside. Actually, however, a film of moisture, dust, oils and other contaminants is usually present in some degree and effectively lowers the resistance of this surface path for electric current.

The resistance also depends upon the geometry of the test *(18)*. In the classical case of two parallel electrodes of area A placed on a solid material of area A and thickness t, the volume resistance R_v would be related to the volume resistivity by the following equation:

$$\rho = \frac{R_v A}{t}$$

For two concentric electrodes with an average perimeter of l and a separation d, the surface resistivity is related to the surface resistance R_s as follows:

$$\sigma = R_s \frac{l}{d}$$

$$l = \pi (r_2 + r_1)$$

$$d = r_2 - r_1$$

$$\sigma = R_s \frac{\pi (r_2 + r_1)}{r_2 - r_1}$$

The unit of resistance and of surface resistivity is the ohm; the unit of volume resistivity is the ohm-centimeter.

The instrument to be described here uses the standard electrode arrangement for measuring the surface and volume resistance of insulating sheets in the electrical industry *(19)*. In applying this principle to fabrics, a fixed electrode arrangement was designed consisting of brass and polystyrene. The total electrode area and the weight of the assembly were chosen to produce the same pressure as is applied to a fabric under the standard test for thickness. For convenience the electrodes were designed to fit a disc with an area of 1/400 of a square yard, as used in fabric-weight determinations. A diagram and two views of the instrument electrodes are shown in Figures 3a, 3b, and 3c. The

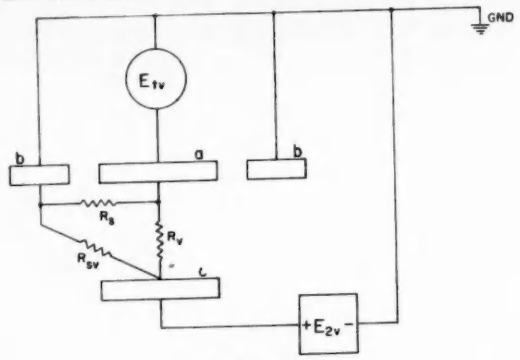

Figure 4
For Measuring Volume Resistance

a = guarded electrode
b = guard electrode
c = unguarded electrode

electrical arrangements and equivalent circuits are shown in Figures 4, 5, 6a, and 7. Figure 6b shows the various components connected for the measurement of surface resistance as diagrammed in Figure 6a and Figure 7. For volume resistance see Figure 4. Under steady-state conditions with neglect of the capacitance associated with each of these resistances, the equivalent circuit would be as in Figure 5.

The following definitions refer to Figure 5 and the volume-resistance test:

R_{shv} = meter shunt
E_{2v} = supply voltage
E_{tv} = electrometer reading
I_{mv} = grid current required by the electrometer
V_a = voltage of point "a" referred to point "b"
V_c = voltage of point "c" referred to point "b"

$$\frac{V_c - V_a}{R_v} = \frac{V_a}{R_s} + \frac{V_a}{R_i} + \frac{V_a}{R_{shv}} + I_{mv}$$

Since the resistance of the power supply is so small compared with the resistance being measured, let

$V_c = E_{2v}$ and $V_a = E_{tv}$

Therefore,

$$\left(\frac{E_{2v}}{E_{tv}} - 1\right)\frac{1}{R_v} = \frac{1}{R_s} + \frac{1}{R_i} + \frac{1}{R_{shv}} + \frac{I_{mv}}{E_{tv}} \text{ ------- } \textit{Equation 1}$$

For measurement of surface resistance see Figure 6a.

The steady-state condition is shown in Figure 7.

The following definitions refer to Figure 7 and the surface-resistance test:

R_{shs} = meter shunt
E_{2s} = supply voltage
E_{ts} = electrometer reading
I_{ms} = grid current required by the electrometer
V_a' = voltage of point "a" referred to point "c"
V_b' = voltage of point "b" referred to point "c"

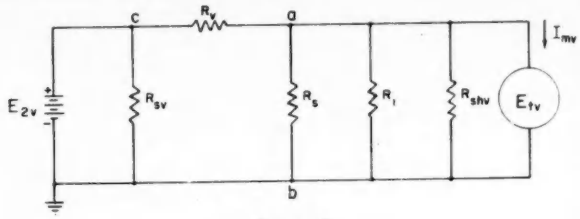

Figure 5
For Volume Resistance

R_v = volume resistance of fabric
R_s = surface resistance of fabric
R_{sv} = leakage resistance from guard electrode to unguarded electrode
R_i = resistance of instrument (about 5×10^{15} ohms)

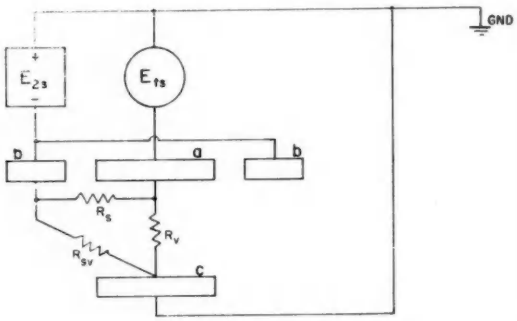

Figure 6a
For Surface Resistance

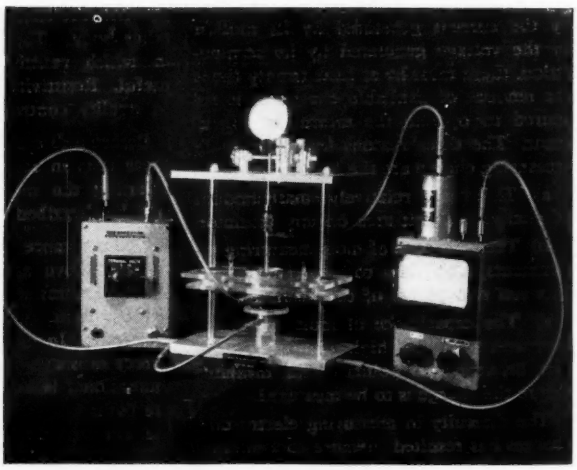

Figure 6b

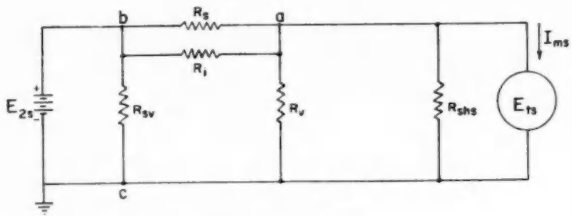

Figure 7
For Surface Resistance

$$\frac{V_b' - V_s'}{R_s} + \frac{V_b' - V_a'}{R_i} = \frac{V_a'}{R_v} + \frac{V_a'}{R_{shs}} + I_{ms}$$

Let $V_b' = E_{2s}$ $\quad$ $V_a' = E_{ts}$

$$\frac{E_{2s} - E_{ts}}{R_s} + \frac{E_{2s} - E_{ts}}{R_i} = \frac{E_{ts}}{R_v} + \frac{E_{ts}}{R_{shs}} + I_{ms}$$

$$\left(\frac{E_{2s}}{E_{ts}} - 1\right)\frac{1}{R_s} = \frac{1}{R_v} + \frac{1}{R_{shs}} + \frac{I_{ms}}{E_{ts}} - \left(\frac{E_{2s}}{E_{ts}} - 1\right)\frac{1}{R_i} \quad \text{------- Equation 2}$$

From Equation 1 and Equation 2, R_v and R_s may be determined in terms of the known quantities, letting

$$\frac{E_{2s}}{E_{ts}} - 1 = K_s \text{ and } \frac{E_{2v}}{E_{tv}} - 1 = K_v.$$

$$R_v = \frac{K_s K_v - 1}{\frac{1}{R_{shs}} + \frac{K_s}{R_{shv}} + \frac{I_{ms}}{E_{ts}} + K_s\left(\frac{I_{mv}}{E_{tv}}\right)} \quad \text{------- Equation 3}$$

$$R_s = \frac{K_s K_v - 1}{\frac{1}{R_{shv}} + \frac{K_v}{R_{shs}} + \frac{I_{mv}}{E_{tv}} + K_v\left(\frac{I_{ms}}{E_{ts}}\right) - \frac{(K_s K_v - 1)}{R_i}} \quad \text{------- Equation 4}$$

For most tests, many of the terms in the denominators of Equations 3 and 4 become negligible.

RESISTIVITIES———

The electrode dimensions

$$r_1 = \left.\begin{matrix}\text{radius of guarded electrode} \\ \text{radius of unguarded electrode}\end{matrix}\right\} = 0.1995 \text{ inches}$$

r_2 = inner radius of guard electrode = 0.3245 inches
r_3 = outer radius of guard electrode = 0.500 inches
Area of center (guarded) electrode = 0.125 inches
ρ = volume resistivity in ohm centimeters
σ = surface resistivity in ohms
Weight of head = 982.4 grams = 1.97 pounds
Total area of electrodes = 0.581 square inches
Pressure = approximately 3.4 pounds per square inch

(The pressure used in standard thickness tests is 3.4 pounds per square inch.)

$$\rho = \frac{R_s (\pi r_1)^2 2.54}{t} = 0.317 \frac{R_s}{t} \text{ (ohm-centimeters)}$$

$$\sigma = \frac{R_s \pi (r_2 + r_1)}{r_2 - r_1} = 13.17 R_s \text{ (ohms)}$$

where t is fabric thickness in inches at this pressure, as measured on the instrument.

The electric field intensity in these tests may be determined by dividing the applied voltage by 0.125 inches in the surface test and by the electrode separation, as shown on the dial gauge, for the volume test. In air at atmospheric pressure, a practical limit to the electric field intensity which can be maintained is about 40,000 volts per inch, and the voltage required to produce this field between the surface electrodes is then about 5000 volts. Applied to the volume resistance electrodes, when a fabric of 0.005 inch thickness is being tested, 200 volts is sufficient to set up an electric field strong enough to cause air breakdown.

Because high field intensities can create corona-type charge leakage and, more significantly, because the resistivities of materials are not ohmic but usually decrease with the voltage applied, measurements of this type should be made with several different voltages applied. Results should be compared with only those made at similar imposed field intensities. Contamination of the atmosphere surrounding the test fabric may seriously alter the effect of high field intensities on the mechanism of leakage. Certain additives lower the breakdown strength of air; and others, like carbon tetrachloride vapor, increase the breakdown strength, allowing higher charge densities for the same geometry.

LIMITATIONS OF THIS TYPE OF TEST———This test measures the specific resistance of a fabric to the flow of a current of charge between two electrodes under the influence of an applied electric field. In actual practice, two other forms of resistivity are important. First is the resistance offered by a fabric to the flow of a layer of surface charge under gradually diminishing charge-density conditions. Second is the resistance between relatively small, oppositely charged, areas. Neither of these types can really be measured by any ordinary resistance test such as this.

A more important limitation to this method lies in its inability to evaluate any other mechanism for charge dissipation except direct conduction. While all antistatic agents seem to lower the resistivity of fabrics, and while most antistatic agents depend upon this lowering of resistivity for their effectiveness, it is possible to enhance one or more of the other types of charge leakage. The resistivity test would not recognize the effect of such an antistatic agent.

Furthermore, it should never be taken for granted that the resistivity is independent of the electric field intensity at the area of measurement. Among dielectrics, resistivities independent of the applied voltage are the exceptions rather than the rule.

For this particular device and test, the terms "surface" and "volume" have been used as they would apply to a homogenous solid sheet. In discussing fabrics,

(Concluded on page P208)

TABLE I

RESISTIVITY DATA FOR TEST FABRICS

Fabric	Description of Treatment	σ = Surface Resistivity, ohms x 10^{12}			ρ = Volume Resistivity, ohms-cm x 10^{12}		
		Nylon	Orlon	Dacron	Nylon	Orlon	Dacron
1	Untreated	20.0	4.9	0.058	5.6	1.7	0.120
2	Treated, Substantive anionic	1.8	0.29	0.017	1.1	0.012	0.042
3	Treated, Cationic antistatic	10.0	1.6	0.010	2.1	0.47	0.021
4	Treated, New type anionic antistatic	0.057	0.0062	0.0029	0.071	0.0045	0.0029
5	Treated, Polyglycol-type antistatic	1.2	0.0057	0.0056	1.1	0.0091	0.028

Measurements were made at 70°C, 65% RH.
Electric field intensity held constant for all materials in surface-resistivity tests.

Rhode Island Section——

NEW NEUTRAL-DYEING METALIZED WOOL COLORS*

H F CLAPHAM

E I du Pont de Nemours & Co, Inc, Wilmington, Del

INTRODUCTION

THE new neutral-dyeing metalized colors for wool and also for nylon are types which many others, as well as I, have waxed enthusiastic about on many occasions. The development of this new type of color probably was concurrent, both in the United States and in Europe, as a patent, US 2,374,106, granted to Kvalnes and Carson of Du Pont, filed in February 1941 and issued in 1945 on the dyeing of nylon with metalized colors without sulfonic acid groups indicates interest of research men in the United States in this type of dye. Dahlen's report on his investigation for the US Army Office of the QM General of 1945 entitled, "The Development of New Dyes and Color Application Processes in Germany and Italy," refers extensively to the chemistry and development of the related Perlon colors in Germany during the early 40's.

Perhaps the first commercially available colors of this type were Polar Grey BL (Geigy) and Acid Alizarine Brown RLL (GDC), introduced as specialty wool colors. At about the same time Du Pont introduced the Capracyl† colors, originally known as Neutracyl colors, with special reference to their usefulness on nylon for extreme light fastness coupled with good wet fastness. While their properties on wool had been evaluated, because of the pressing problems of fastness on nylon at the time, practically all of the emphasis was placed on their value on that fiber, and their particular adaptability to wool was only partially recognized.

As a matter of fact, some of the earliest uses on wool resulted from their evaluation as colors for wool-nylon blends, particularly in the field of military fabrics.

Evidence of the considered value of this new type of dye is attested by the introduction of related lines by a number of dye manufacturers, such as Irgalans, Violans, Cibalans, Chromolans, Supralans, etc, during the past several years.

As a technical representative of the Du Pont Company, it is natural that my knowledge and experience in this type of dye is most intimate with the Capracyl dyes. The normal testing of competitive types has led to some knowledge of the related dyes mentioned above. This testing indicates that, while there are the usual differences in properties from line to line and dye to dye, the general group has related properties. I shall devote the remainder of this paper to a discussion of the application and fastness properties of the Capracyl dyes, with which I am most familiar.

H F Clapham

Neutral-dyeing metalized dyestuffs that do not contain active sulfonic acid groups constitute a comparatively new class, which is useful in the dyeing of fast shades on both wool and nylon. The author reports that the development of these dyestuffs took place concurrently in Europe and the United States and that the full usefulness of such products was not at first appreciated. He discusses the fastness properties of the Capracyl brand of these dyestuffs on wool and nylon, points out how simply they are applied, and presents experimental curves showing exhaustion rates on wool and nylon.

GENERAL PROPERTIES

The Capracyl dyes are neutral-dyeing metalized types, as indicated in the title of this paper. As previously stated, they resulted originally from a search for dyes for nylon with outstanding fastness properties, especially fastness to light. Subsequent testing and actual use have shown that they possess equal or perhaps even greater value for the dyeing of wool. This class of dye is characterized by relatively dull shades and a lack of good bright or medium-blue and green representatives. For this reason, we recommend the use of Du Pont Anthraquinone Milling Blue BL and Du Pont Anthraquinone Green GNN to supplement the line. The application and fastness properties of these two types make them compatible with the general range of Capracyl dyes. A high general level of fastness coupled with a high degree of light fastness indicates particular usefulness for men's wear, auto upholstery, carpets, and similar related materials where use requirements dictate a high degree of fastness. I was told by several outstanding woolen-mill dyers and chemists that this new class of wool dyes will probably gradually replace many of the chromate-dyeing types for light-to-medium shades because of ease of application and excellent fastness properties. As an example of this, it is possible to duplicate the shade of a well-known chrome-dyeing brown type with fully equal wet fastness and perhaps even slightly superior light fastness with a combination of Capracyl colors. Similarly, Capracyl Gray GN has fastness comparable to a well-known chromate-dyeing gray with somewhat better spotting fastness.

LIGHT FASTNESS ON WOOL AND NYLON——It might be well to mention at this point that the Capracyl line does require some judgment in selection of combinations, especially for wool. Because they were first developed for nylon, three dyes are included in the range that are particularly fast on nylon but have relatively poor properties on wool, as compared with the remainder of the group. These are Capracyl Brown RD, Blue G, and Black N. The remaining types are equally useful on both wool and nylon. This is illustrated in Table I showing Fade-Ometer ratings for dyeings of all of the color types on both wool and spun nylon.

TABLE I

FADE-OMETER LIGHT FASTNESS

	Ratings Only	
	Wool	Nylon
Capracyl Yellow NW	6	7
Capracyl Yellow 3RD	6	7
Capracyl Orange R	6	6
Capracyl Brown RD	4-5	5
Capracyl Red B	6	7
Capracyl Red BB	5	4
Capracyl Violet R	5	5
Capracyl Blue G	3	5
Capracyl Gray GN	4	4
Capracyl Black N	8	8

* Presented in the quarters of the Providence Engineering Society, Providence, R I, on March 27, 1953. Ms rec'd by the Editor of Proceedings on February 4, 1954 and submitted to the Reporter on Feb 9, 1954.

† Capracyl is a registered trade-mark of E I du Pont de Nemours & Co, Inc.

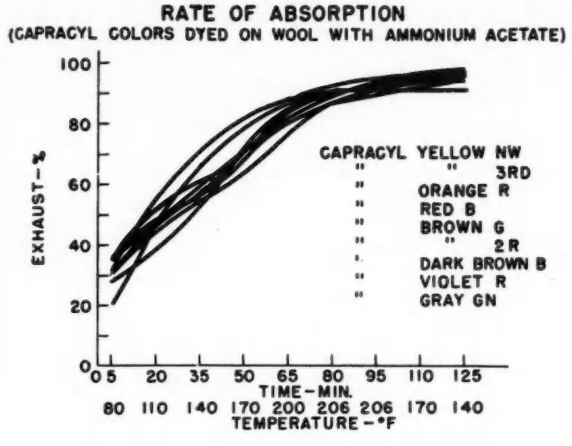

Figure 1

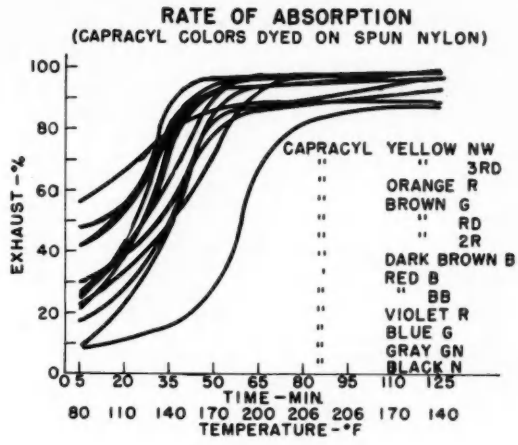

Figure 1a

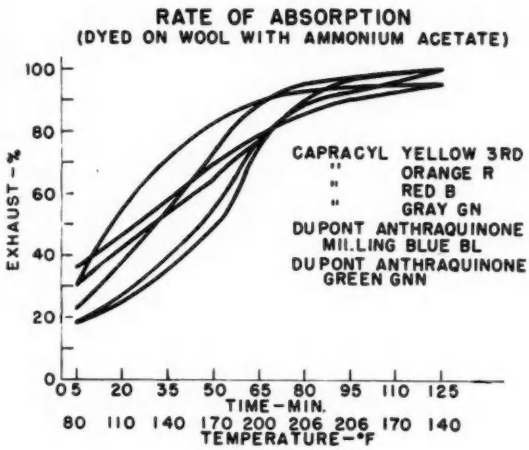

Figure 2

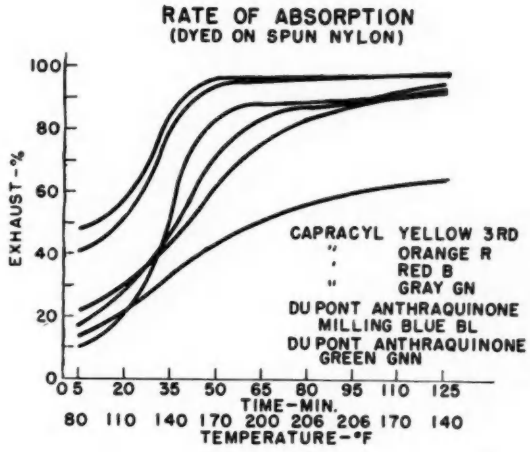

Figure 2a

Extensive light-fastness testing of a wide variety of shades made up with combinations of Capracyl colors supplemented with Du Pont Anthraquinone Milling Blue BL and Du Pont Anthraquinone Green GN against similar shades made with previously available color types has shown that equal and usually superior fastness results from the new group. Of particular interest is the fact that, practically without exception, a monotone fade results with the Capracyl combinations. As you all know, this is a basic aim in any formulation where light fastness is a factor.

CAPRACYL DYEING METHOD—— As previously stated, ease of application is one of the outstanding virtues of this new class of dyes. The Capracyl dyes are neutral-dyeing and, as might be expected, require little or no chemical aids for exhaustion. The usual general procedure for the application of Capracyl dyes is to wet out the wool with 1-2% conc ammonia water owf (on weight of fiber), to add the dye at 100-120°F, to raise the temperature slowly to the boil, to run 30 minutes, to add 1-5% ammonium acetate or 1-3% acetic acid 28% for exhaustion, if required, to boil 15 minutes and finally to sample. On rawstock and top, the above method is adequate in most cases to obtain level, well-penetrated dyeings. Where a shade is being matched and additions are likely to be required, it is probable that, wherever possible, the acetic acid or ammonium acetate should be omitted so that the addition can be put on in a neutral bath at 150-170°F without undue unlevelness.

PIECE DYEING——Piece dyeing with this type of dye requires more care in application to insure maximum levelness. While these dyes do not transfer to any great degree at the boil, they do cover well and, if approximately the same precautions are used as for chromate dyeing, level, well-penetrated pieces can be produced. As standard practice on piece goods, we would recommend wetting out the goods with 2% of conc ammonia water, adding the dye at about 100°F, raising the temperature slowly to the boil, and boiling for 30-45 minutes. Anhydrous sodium sulfate is of no value in the application of this type of dye and, in fact, may have a slightly undesirable effect on the solubility of the dye. Acid or acid salts are not recommended, especially where feeds of dye may be required, as they would cause too rapid exhaustion and consequent unlevelness. It is possible, however, that acid may be required for certain darker shades to obtain the most economical exhaustion, but great care should be taken in its use to insure satisfactory coloration. Retarding agents are available and are discussed later in this paper.

EXPERIMENTAL

ABSORPTION CURVES —— Many data have been accumulated on the rate of dyeing of this class of dyes on both wool and on nylon by use of the tech-

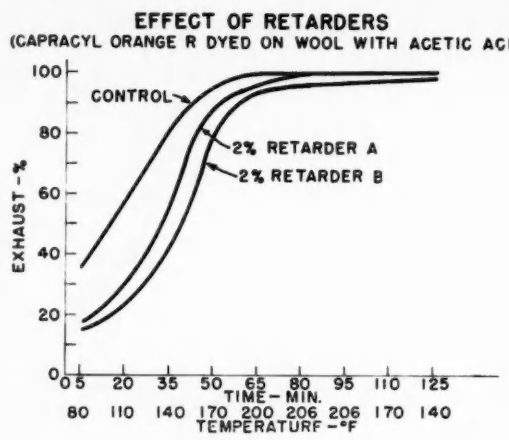

Figure 3

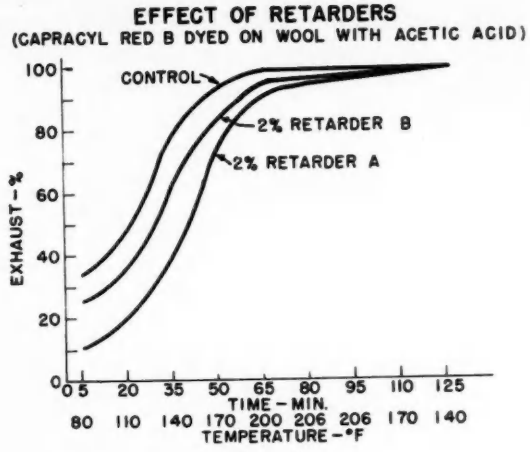

Figure 4

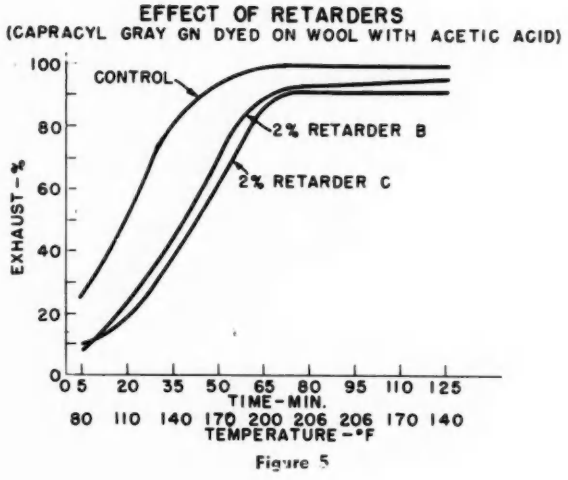

Figure 5

niques described in the Philadelphia Intersectional Contest Paper of 1950. In Figure 1 we show curves of the rate of dyeing of all the Capracyl colors that are suitable for wool dyeing. We have purposely put them all on one plot so that you may see that all members of the class have relatively similar dyeing rates. The dyeings represented by these curves were made with 5% ammonium acetate owf in the dyebath. In Figure 2 we show several Capracyl types along with Du Pont Anthraquinone Milling Blue BL and Du Pont Anthraquinone Green GNN to illustrate the compatibility of the dyeing rates of these two supplemental colors to the Capracyl range. As in Figure 1, these curves were plotted from dyeings made with 5% ammonium acetate. Figure 1a and 2a show similar data plotted for spun nylon. You will see that there is a wider spread for nylon than for wool.

LEVELNESS TESTS —— Since the Capracyl colors are neutral-dyeing types and because levelness is always an important consideration in any dyeing, tests of this important property have been made. The usual skein-to-skein transfer test at the boil shows only a relatively low degree of transfer on a 45-minute boil. It is, in general, on the order of 25% transfer, not enough for good leveling. The dyes do, however, show remarkably good covering power and rate with the best chrome dyes in covering mixed wools uniformly. These facts, coupled with a properly selected dyeing method, make fully satisfactory dyeings possible.

Laboratory research has indicated that there are several available retarding agents for this class of dye. Nonionic wetting agents as a class exert a pronounced retarding effect. Figures 3, 4, and 5 show data obtained in the same manner as for the rate-of-dyeing curves previously shown. You will note that in each case Retarders A and B, C and D all show marked retarding in the early stages of the dyeing, but that the final exhaustion is practically equal to that of the control dyeing. These agents give little, if any, releveling effect but work only to cause a more gradual deposition of the color with accompanying more uniform and level results during original dyeing.

Similar data, plotted for spun nylon show a similar retarding effect.

While the tests are not complete as yet, there are strong indications that the use of nonionic retarding agents may seriously affect the light fastness of several Capracyl-type dyes.

STRIPPING——Stripping is always an important consideration, both as a means of correcting mistakes or meeting style-change requirements as well as for clearing stocks of reworked or reused wool. Tests indicate that the Capracyl colors can be stripped to very light shades by the following technique:

Boil off with 5% ammonia water (26%) owf for 20 minutes at 200°F and rinse.

In a fresh bath add 3% formic acid, 85%, raise the temperature to 160°F, add 4% Sulfoxite S Conc, raise the temperature to 200-205°F and run 45 minutes.

Rinse and neutralize with 3% ammonia water (26%) at 120°F for 20 minutes.

Generally speaking, the initial ammonia boil-off strips between 20 and 40% of color from the dyeings.

DISCUSSION OF FASTNESS PROPERTIES

The fastness properties of the Capracyl colors have been mentioned broadly in the preceding portions of this paper. Perhaps a somewhat more detailed examination of the more important fastness properties will serve further to emphasize their value.

It has already been made clear that light fastness is generally of the highest order and lends itself to those uses where light fastness is of paramount importance.

Fastness to wet processing through the

(Concluded on page P208)

1953 Annual Convention
Cotton Group——

PRACTICAL PARTIAL ACETYLATION OF COTTON*

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ESMOND J KEATING, and CHARLES F GOLDTHWAIT

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INTRODUCTION

EARLIER reports from the Southern Regional Research Laboratory (*1-3*) on fibrous, partially acetylated cotton have been primarily concerned with preparation of materials on a sufficient scale to permit evaluation of their properties and with a presentation of the results obtained. These have indicated very desirable improvements in some cotton properties, principally heat and rot resistance. The potentialities of the acetylation process for producing a better fabric than is afforded by the usual additive finishes were believed to justify the development of semipilot-plant-scale methods for its commercial manufacture. This has been accomplished, and the methods have been adopted by companies who are now in production of partially acetylated cloth; and, in addition, these methods are being actively considered by others for cloth, yarn, and raw-stock acetylations as well. In this paper, the practical aspects of current partial acetylation processing will be emphasized.

CHEMICAL NATURE OF THE PRODUCT——With the proper catalysts and conditions, all of the hydroxyls of the cellulose that composes cotton can be esterified through reaction with acetic anhydride, that is, the hydrogens of each hydroxyl on the anhydroglucose units can be replaced by acetyl groups. In the practical application of fibrous acetylation, it is unnecessary to substitute with 3 acetyls on each glucose unit (DS = 3) throughout the entire cotton fiber. Substitution at about one-third, or even less, of all the sites is adequate, the main requirement being acetylation of the more readily accessible hydroxyls in the amorphous regions and crystallite surfaces (*4,5*). The product is believed to be a structure of crystalline cellulose with interspersed cellulose triacetate, averaging nearly one acetyl per glucose unit (21% acetyl by weight) through the entire mass. This composition will be used as a basis for the discussion of fibrous partial acetylation. Fibers of cotton acetylated to DS = 1 by the methods described almost invariably appear uniform when dyed and examined with the microscope. There is nothing to suggest a distribution of acetyls anything like a protective layer or sheath around each fiber. A full discussion of the nature of these products has already been published (*6*).

The degrees of substitution of acetate rayons and partially acetylated cottons may overlap, but their properties are greatly different. Even among acetylated cotton products which have retained their fibrous nature throughout processing, there are great differences between products obtained by preparing partial acetates approached from the cotton side, as opposed to those prepared by deacetylation, or saponification, from a higher value, as illustrated in Table I. It is seen, for example, that with two products of the same average composition, approximately one-third substituted, the one prepared by acetylation up to that value behaves almost like the nearly completely substituted product in dyeing, water retention, and rot resistance; the other, prepared from a higher substituted product by deacetylation, dyes, retains water, and rots very much like ordinary cotton (*6*).

E M Buras, Jr

Fibrous partially acetylated cotton was first prepared in England about 1900, but practical applications of outstanding properties of this material, heat and rot resistance, have been developed in the United States only recently, following the development of practical processes for its preparation at moderate cost. The Southern Regional Research Laboratory has worked out both batchwise and continuous processes. Fabric can be handled on stainless steel dye jigs, as used in cotton finishing plants, or on a newly developed continuous range; yarns may be treated in package form or continuously as warp; and raw stock may be handled in a raw stock dyeing machine. Well adapted for commercial use, the processes employ acetic acid and anhydride, with small amounts of perchloric acid as catalyst, to convert cotton into a new type of textile fiber. Data on the concentration of chemicals used, duration of treatments, temperature, and other conditions required for best results have been determined. Several commercial developments are based on these processes.

ACETYLATION PROCESSING

Preliminary purification is preferred for both batchwise and continuous acetylations of yarns and fabrics, since non-

TABLE I

EFFECT OF ACETYLATION AND DEACETYLATION ON PROPERTIES OF COTTON

Sample acetyl	DS	Dyeing	Swelling Capacity	Strength after burial		
				0 days	14 days	6 months
%		**	*** %	%	%	%
0	0	blue	38	100	0	0
23	1.1	yellow	23	100	100	100
41	2.6	yellow	20	106	104	98
23*	1.1	blue-green	32	100	0	0

* Deacetylated from DS = 2.2, 37% acetyl.
** A direct blue and an acetate yellow in same bath (*2*).
*** Water held on centrifuging at 5820 times gravity (*7*).

* Presented by E M Buras, Jr at the Conrad Hilton Hotel, Chicago, Ill, on September 18, 1953. Ms rec'd by the Editor of Proceedings on Jan 27, 1954 and submitted to the Reporter on Feb 2, 1954.

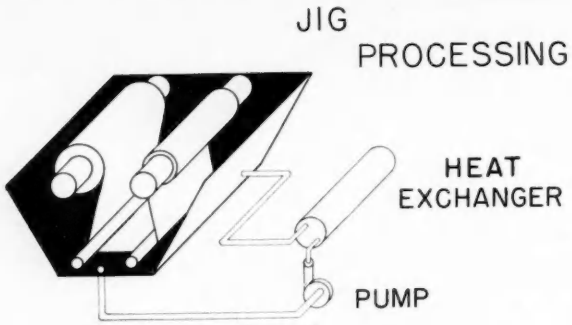

Figure 1
Diagram—jig acetylations

Activation—glacial acetic acid, 1 hr, room temp
Reaction—85% glacial acetic acid, 15% acetic anhydride, 0.15% perchloric acid, 1 hr, 68°F
Wash—water

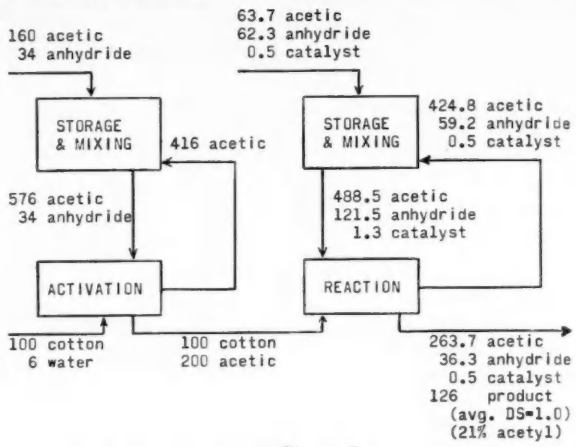

Figure 2
Flow sheet—jig acetylations (relative weights)

cellulosic materials in or on cotton may interfere with its esterification. This is done by desizing and then boiling for 2 hours in 2% caustic soda solution containing a wetting agent. The cotton must be given an acetic acid sour and well rinsed to avoid getting free alkali or salts into the reaction mixture where they could neutralize the catalyst. The cotton is then dried. In the batchwise acetylation of raw stock, preliminary purification is omitted.

BATCH PROCESSING —— The procedure in use at the Southern Regional Research Laboratory has 3 principal stages: activation, esterification, and washing (Fig 1). The first step, activation of the cellulose, is accomplished by a preliminary soaking of the cotton in glacial acetic acid at room temperature for a minimum period of 1 hour, although the time may be extended to overnight, if more convenient, without harm to the cotton. After activation, the acetic acid is drained or squeezed from the cotton.

The esterification is effected by treatment with a mixture of 85% acetic acid, 15% acetic anhydride, and 0.1-0.2% perchloric acid based on the mixture, which totals about 8 times the weight of the cotton. The reaction mixture is maintained at about 68° F. Besides initial cooling, there is to be removed heat resulting from the esterification reaction and heat transferred from the jig and surroundings, so an external heat exchanger is used. Batches of 100 to 200 pounds of cotton are usually acetylated at a rate of 60 pounds per hour. The speed of reaction is conveniently regulated by piecemeal addition of the anhydride. Pronounced differences in rate of acetylation are sometimes found, due to differences in nature, weight, construction, and degree of purification in the cotton materials being treated. To evaluate these differences for purposes of control, trial acetylations are often made *(2)*, with test dyeings being made at frequent intervals during the reaction. The length of time of acetylation is then adjusted accordingly for the larger-scale operations.

For most commercial uses, a sufficiently uniform product is obtained from one run to the next by duplication of times, temperatures and bath compositions. Small trends upward or downward, noted upon chemical analyses or test dyeings of samples from each completed roll while the next is being processed, are readily corrected by small changes in the duration of treatment, or concentration of catalyst, or both. Sufficiently precise analyses of the baths for their principal components may be performed with relatively simple apparatus and rapid methods devised at this Laboratory *(8)*.

After the reaction is completed to the extent desired, the fabric can be squeezed to recover much of the solution for re-use. If properly replenished, the solution can be used as many as 10 times before setting aside for recovery. Final treatment consists of washing and drying the fabric in a conventional manner. It is usually possible to recover full width in fabrics with no loss in length.

The generalized process described above has been carried out at our Laboratory on lots of up to 200 pounds of cotton *cloth* of weights in the range of 2 to 10 ounces per square yard on a 50-inch stainless-steel tensionless dye jig similar to those in common use, except that it is hooded and equipped with an exhaust system and an external heat exchanger. Acetylation of a typical 100-pound roll of 6-oz sheeting is accomplished in 1¼ to 1½ hours at 68° F. This scale of operations has required a 3-ton rate of refrigeration to maintain this temperature under local conditions.

In order to get greater utilization of a single jig, a separate tank may be used for the pretreatment. The fabric, after purification, is rolled up with minimum tension and immersed in the tank filled with acetic acid-anhydride mixture for the activation.

The economy of the process depends upon the re-use of solution, and the complete flow sheet is shown as Figure 2. The replenishing of reagents is done in the storage tanks ahead of the next run to avoid that source of heating within the jig.

The net over-all materials balance, given in Table II, is typical of current practice. About 2 pounds of acetic acid, 1 pound of acetic anhydride, and 0.005 pound of perchloric acid are used for each pound of cotton converted. The combined cost of these chemicals at current market prices is about 40 cents.

In *yarn* acetylations over the past 8 years at the Southern Regional Research Laboratory, several hundred pounds in small lots of various kinds have been successfully treated on a 6-pound package dyeing machine adapted for acetylation by the addition of coils and ventilation. The solutions and times of treatment are substantially the same as in jig processing. Installation and adaptation of a 50-pound combination raw-stock and package dyeing machine is in progress.

TABLE II

NET OVER-ALL MATERIALS BALANCE—JIG ACETYLATIONS

(Typical current SRRL practice)

	IN	*OUT*
	lb	*lb*
Fabric	100	126
Acetic acid	200.6	—
Anhydride	119.4	—
Moisture in cloth	6	—
Catalyst	0.5	—
Liquor carried out	—	300.5
Total	426.5	426.5

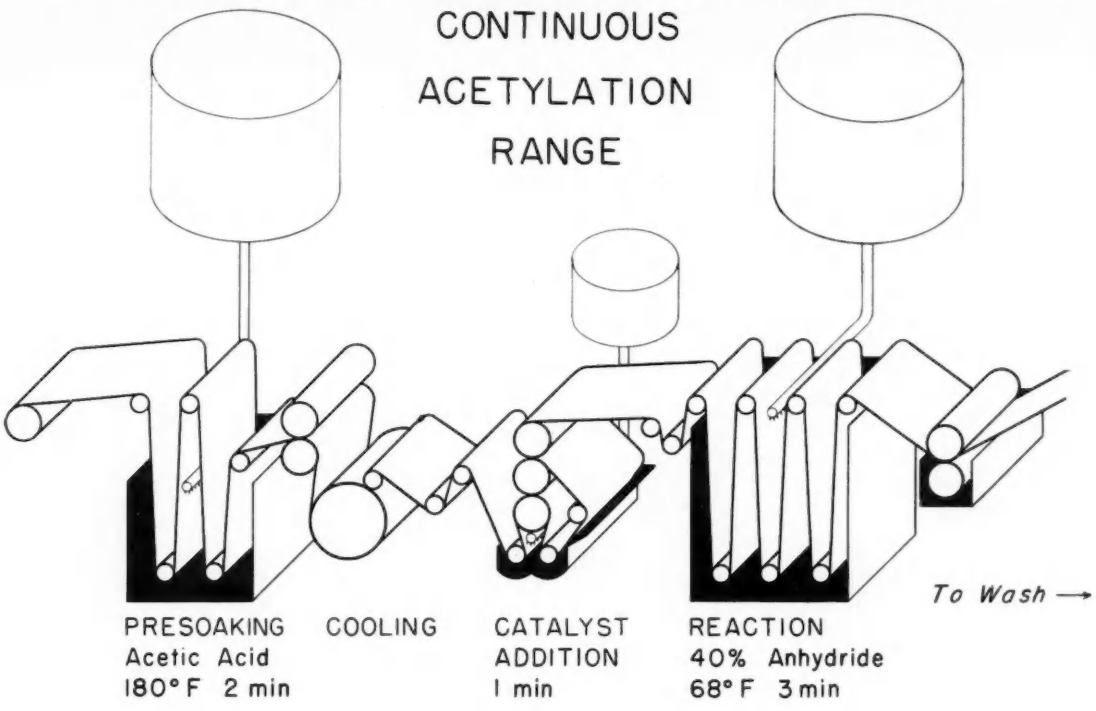

Figure 3
Diagram—continuous acetylation range

For preparation of acetylated *raw stock* at this Laboratory, several bales of cotton have been similarly treated in a 50-pound-capacity stainless-steel kier. Yarn from such acetylated raw stock has been spun on standard cotton processing equipment in which the count-strength product is only a little less than would be expected considering the weight added to each fiber by acetylation *(9)*. Not all problems connected with mechanical processing have been solved, however. Investigations are under way to improve further the processing qualities of acetylated raw stock through variation in chemical processing techniques and subsequent addition of antistatic agents. The basic steps of the acetylation treatment as presented here will apparently be retained.

This generally described process has been adopted commercially. *One* company is in production on a 70-inch jig. Three-shift production is about 2000 yd of 57-inch, 7-ounce-per-square-yard cloth. A *second* company is producing on two wide jigs. These companies began production over a year ago, and their products are now available for the commercial laundry trade and at department stores in the form of scorch-resistant home ironing-board covers. A *third* company has experimentally acetylated several hundred pounds of raw stock. Others are conducting independent development of raw-stock, yarn, and fabric acetylation processes based largely on information furnished by the Southern Regional Research Laboratory.

CONTINUOUS PROCESS —— The continuous process is based on the same reaction as the batchwise process, but the reaction is more rapid and is accomplished as shown schematically in Figure 3. The textile material is entered into the machine at normal regain. The first bath is glacial acetic acid at 180° F. This accomplishes the activation in about 2 minutes instead of 1 hour as in the batch process. The cotton must then be cooled. This may be done as a second step by letting part of the acid evaporate, thereby cooling the remaining materials or, more economically and uniformly, by passing the textile material around a cooling can through which water is circulated.

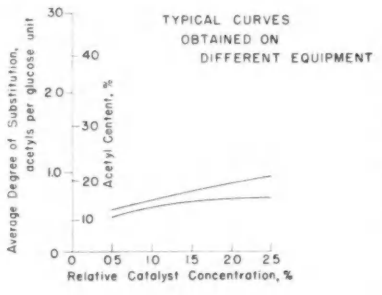

Figure 4
Effect of catalyst concentration on extent of reaction

The third step is catalyst addition. As distinguished from batch processing, however, the catalyst is kept separate from the acetic anhydride. By maintaining a concentration of 1 to 2% perchloric acid in an acetic acid bath, the subsequent esterification is speeded up sufficiently. Figure 4 shows the effect of changes in catalyst concentration on the extent of reaction. It may be seen that, while this factor may be used to govern the extent of reaction, the system is not undesirably sensitive to small changes over the commercial range of substitution. It may be noted in consideration of the catalyst addition step that the primary factor in determining the extent of reaction of the cotton is the *concentration* of perchloric acid, rather than the total pickup. Solution that is outside of the fiber itself comes off in the next bath. Only the catalyst retained in the porous structure of the cotton affects the reaction, and this retention is relatively unaffected by subsequent mechanical operations. Thus, unevenness of catalyst roll pressure is not as serious a problem as in additive finishes where the total quantity of additives is dried into place. Since separate application of the catalyst provides for close control of the extent of reaction and of uniformity, substantially standard processing equipment and standard techniques can be used.

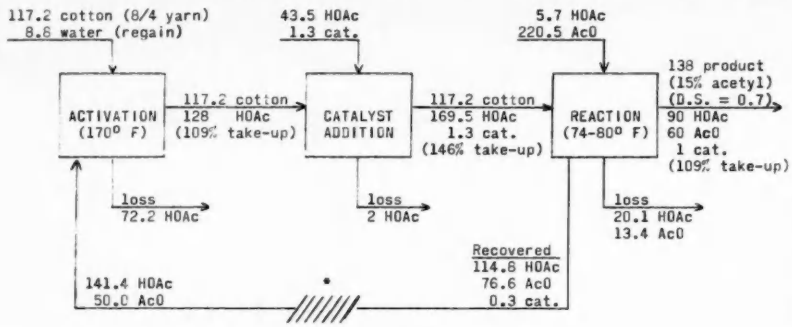

Figure 5

Flow sheet—continuous processing

All weights in pounds.
Losses due to sampling and evaporation.
Abbreviations: HOAc = acetic acid; AcO = acetic anhydride; cat = perchloric acid catalyst.

* Recovered solution could be cycled if excess acetic anhydride and catalyst were removed. For this run, the activation tank was fed with fresh reagents and, for the cost estimate in the text, is considered as requiring no additional chemicals.

The fourth step provides the final reagent, acetic anhydride, in sufficient quantity by immersion of the fabric. The reaction proceeds to practical completion in about 1 to 3 minutes at 70-75° F, depending also on the anhydride concentration in the acetic acid medium.

The final operations are washing and drying, conducted in units of standard design.

The first model continuous acetylating machine with continuous feed tanks and hoods installed is 35 ft long and is designed to process 15" fabric or warp of 200 ends of #1's yarn or the equivalent. It has been found possible to acetylate both yarn and fabric at speeds from 3 to 20 yards per minute, depending on the construction and degree of purification. Hence, commercial application has taken the direction of full-scale adaptation of that apparatus. Assembled from commercially available pads and similar range-type units, a full-width machine has been built and installed for commercial production. Various fabrics are being processed experimentally on this machine.

Estimation of the over-all cost of producing partially acetylated cotton is beyond the scope of this paper. Factors, such as cost of development, amortization and overhead, promotion, shipping, taxes, and profit must necessarily be estimated by the commercial producer. Auxiliary devices necessary for safety or to abate fumes cannot be considered in detail until a specific location is planned.

The direct chemical costs, however, can be estimated from an examination of a typical flow sheet and materials balance (Figure 5) obtained from a continuous run on the model machine described above. Re-use of the recovered solution for the activation step requires removal of excess acetic anhydride (beyond that required to react with regain moisture) and perchloric acid. One hundred twenty-six pounds of a warp of 496 ends of 8/4 yarn was acetylated at 3.71 yd/min through the use of approximately 43.5 pounds of acetic acid, 226.2 pounds of commercial-grade acetic anhydride, and 2.2 pounds of 60% perchloric acid. The net chemical cost, at 12¢ per pound for acetic acid, 15¢ per pound for acetic anhydride, and $1.10 per pound for perchloric acid, is therefore about 33 cents per pound of cotton treated. Of course, items of expense due to evaporation and waste may assume greater importance in larger-scale operations, and more extensive data would be required to obtain accurate cost estimates on cotton materials of different construction and weight.

An economy could be effected through recovery of materials now washed out and discarded, but no data have been obtained in the pilot plant. The use of a hot-flue dryer should be quite feasible for recovery of the major part of the carryout from the last bath, if steps were taken to first neutralize the catalyst, but this has not yet been demonstrated in practice.

POTENTIAL USES

Some of the major potential uses for partially acetylated materials are listed in Table III along with the latest cotton consumption figures in pounds *(10)*. It is believed that, for a substantial part of these uses, cotton could be partially acetylated to give products which could be in a better competitive position than cotton itself on a basis of cost per unit of service life. While it would not be expected that all materials in these categories would be replaced by partially acetylated cotton, these figures nevertheless indicate great possibilities.

TABLE III

POTENTIAL ACETYLATED COTTON USES WITH 1951 UNITED STATES COTTON MARKET

End uses	*Cotton market** *478-lb bales*
Auto tops, convertible	2,720
Awnings	43,720
Barricade and sand-bags	—
Coated truck fabric	6,250
Conveyor belts	17,230
Diapers, collection service	—
Filter fabrics	20,570
Flags	4,200
Flat-work-ironer aprons	10,250
Flat-work-ironer cover cloths	26,870
Flat-work-ironer feed ribbons	420
Flat-work-ironer padding	10,600
Home-ironer covers	—
Home-ironing-board covers	—
Hot mill gloves	6,730
Industrial thread	142,600
Insulation, electrical	130,210
Insulation, thermal	150
Laminate fabrics	—
Net and seine twine	58,680
Paper-dryer felts	—
Press-bed covers	10,930
Press-bed flannels	24,240
Press-bed padding	12,020
Shoe linings, leather	62,300
Shoe fabrics, rubber	40,440
Shower curtains	890
Tarpaulins	93,640
Tents	35,290
Umbrellas, beach and garden	1,480
Water-softener bags	—
Zipper tape	8,080
TOTAL	770,510

* Latest National Cotton Council of America revised estimates *(10)*.

PRODUCT EVALUATION——Undyed cotton cloth and yarn partially acetylated to DS = 0.7 to 1.0 is unchanged in color, is odorless and nontoxic, and is unchanged in appearance. Higher degrees of acetylation do, however, produce some stiffness. A number of other properties are decidedly changed. First, and outstandingly so, acetylated cotton is highly resistant to attack by microorganisms. Typical results obtained on burial of rotproofed cotton fabrics in highly active soil are shown in Figure 6. Under the conditions used at the Southern Regional Research Laboratory, untreated cotton loses practically all of its strength in one week; fabrics treated with copper-containing additives are reduced to 30% of original strength in 8 to 12 weeks; and properly acetylated goods retain 80 to 100% strength for longer than 50 weeks. Actually, a small gain in strength is often observed in acetylated cotton during the early stages of these burial tests; this effect is paralleled in heat tests as well (Figure 7).

Secondly, and of great commercial interest, is the heat endurance of fibrous partially acetylated cotton materials. Figure 7 gives typical heat-endurance figures. In this graph is shown the strength of acetylated yarn for oven exposures at two different temperatures plotted against time on a logarithmic scale. At 250° C (approx 400° F) cotton is reduced to 2/3 strength in 3 minutes,

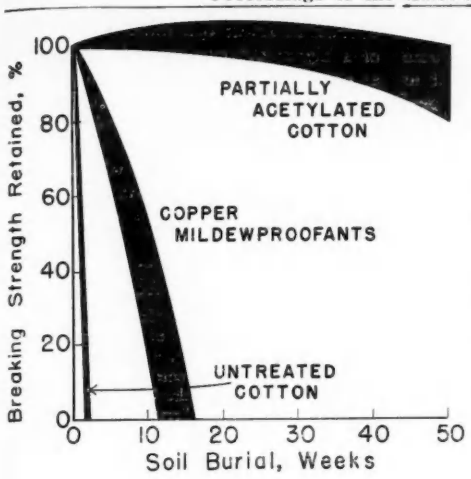

Figure 6
Typical results of SRRL burial tests on treated cottons.

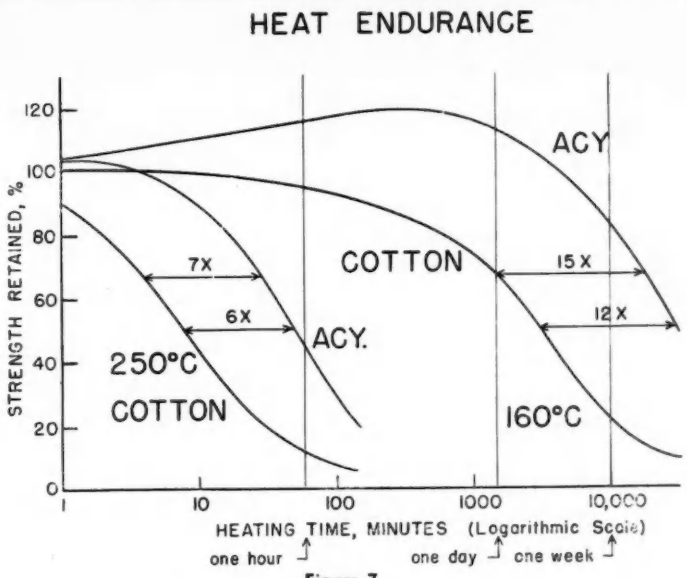

Figure 7
Heat endurance of partially acetylated cotton.

while comparable yarn made up from acetylated raw stock is down to 2/3 strength only after 25 minutes, about 8 times as long. Similar comparison at the ½-strength criterion gives acetylated cotton a 6 times advantage. Likewise, at 160° C (approx 320° F) acetylated cotton yarn lasts 12 to 15 times as long as ordinary cotton. It is not surprising that, in laundry-press-pad covers, acetylated sheetings give 4 to 5 times the life of cotton canvas *(2)*.

Other outstanding contrasts of acetylated products with their natural cotton counterparts are lower moisture regain and resistance to certain types of chemical attack. For example, the normal regain of acetylated goods is about 3 to 4 percent, and it has been found that, in 20% hydrochloric acid, acetylated yarns lose only 33% strength in 8 hours, roughly half as much as untreated cotton *(11)*.

As to weathering, acetylated cotton materials have generally proved to equal cotton. Current investigations are showing, however, that acetylated cotton may have outstanding weather resistance. For example, certain samples dyed prior to acetylation and recently exposed in New Orleans showed 78% strength retention after one year of outdoor exposure. Untreated and dyed control samples each retained only about 30% strength over the same period *(12)*.

CONCLUSION

The acetylation process described in this paper has been developed through semipilot-scale operations on batchwise and continuous bases to the point of commercial production of fabric, and sample production of yarns and raw stock. The value of partially acetylated cotton for various end uses has been demonstrated in practical trials. It is hoped that the progress reported here will not leave the impression that process research is complete, but rather, that sufficient progress has been achieved to make possible satisfactory semipilot-scale operations and several installations of commercial importance.

ACKNOWLEDGMENTS

The writers are greatly indebted for innumerable direct contributions by other members of the Cotton Chemical Processing Section; thanks are also due to Cotton Mechanical Processing Section for materials, and the Analytical, Physical-Chemical, and Physics Section for many tests.

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DISCUSSION

Question: What is the effect of partial acetylation on the physical properties of a cotton fabric, especially on its tensile strength?

Answer: It does not have a pronounced effect on the physical properties, and it does not impair the final tensile strength on the average more than two percent. Because the strength of the swollen fabric during acetylation is reduced about 20 to 30%, however, it is important that the process of partial acetylation be carried out on equipment that protects the fabric from distortion.

Q: Have you tried disperse (acetate) dyes on partially acetylated cotton?

A: Yes, it dyes uniformly with disperse dyes when the partial acetylation is uniform. A good test for uniformity is to dye the treated cotton with a mixture of a yellow disperse dye and a direct blue cotton dye. A uniform color indicates uniform partial acetylation.

Q: Does vat dyeing appreciably deacetylate partially acetylated cotton?

A: A number of exploratory vat dyeings have been made on partially acetylated yarns in the laboratory, and surprisingly little effect was detected on the rot resistance of the yarn, as determined by soil burial. However, each possible application requiring vat dyeing should be considered as a special case, inasmuch as only a very little deacetylation can be tolerated.

Q: How do you account for the great difference in reaction time for partial acetylation between the batch method and the continuous method?

A: The concentration of catalyst for the continuous method is about ten times as great as for the batch method, the temperature is higher, and the concentration of acetic anhydride is increased. By the nature of continuous methods, however, uniform results over the cotton can

nevertheless be attained. In batch processing, satisfactory control is difficult if the speed of acetylation is as fast as in the continuous method.

Q: Will you please repeat how long it takes to prepare package yarn for acetylation?

A: Desizing and alkaline boiling of cotton yarn can be accomplished in one to 1½ hours according to the yarn, the machine and local conditions. Activation of yarn in glacial acetic acid at room temperature requires at least one-half to one hour. Esterification then takes one-half to one hour. Washing and drying complete the process for a total elapsed time of about 4 hours.

Q: Can the concentration of catalyst be made too high?

A: In continuous work acetylation is effected in about one minute by the use of a higher concentration of catalyst, etc, as previously described. In batch processing, however, such rapid acetylation cannot be satisfactorily controlled to attain uniformity from batch to batch, or even throughout one batch, whether the processing is carried out on piece goods in a jig or on yarn in a circulating machine.

Wool Colors——

(Concluded from page P202)

mill is also an important consideration, and the Capracyl colors in light-to-medium shades show good resistance to the normal woolen- and worsted-mill processing. They are on a par with the usual chromate-dyeing colors in this respect.

Carbonizing fastness is always an important consideration. Tests of this property indicate that in medium shades there is little effect on shade, no effect on light fastness, and perhaps a slight lowering of wash fastness. In pastel colors, carbonizing causes considerable change in shade. As a slight scour brings the shade back, Capracyl dyes are really about equal to the usual chromate dyes in carbonizing fastness.

Tests indicate that decating causes some weakening of shade with little or no effect on general fastness.

Chlorination for shrink resistance on woolens causes a slight reduction in light and perspiration fastness. The effect on shade varies from color to color, and several of the dyes appear to have interesting properties on materials that will be subject to the chlorination type of shrink-resistant treatment.

We have stated that the end-use fastness of these dyes compares very favorably with the best of the older types of wool colors on the market. In the case of light fastness the results are practically always superior to combinations of the older types.

In conclusion, I would like to suggest that the Capracyl class of neutral-dyeing metalized dyes, offers a novel and useful tool to the dyer for the production of fast shades with a minimum of processing time and chemicals. These savings in time and chemicals are not only of value in themselves but have the side result of less damage to the wool fiber with consequent better all-round finished products.

—♦ ♦—

Resistivity——

(Concluded from page P195)

the "surface" should be considered as that area in actual contact with an electrode of a definite size under a defined normal load. "Volume" should be considered as that part of the fabric between a top and a bottom "surface". The relationship between the surface and volume of the fabric and the surface and volume of the yarns and fibers is obviously complicated. Fortunately, however, for most of the applications where fabric resistivity is important, it is unnecessary to explore this relationship, since this type of measurement is no better than the simplified concepts of surface and volume given above.

EXPERIMENTAL RESULTS —— Thoroughly scoured and dried fabrics of Orlon, nylon, and Dacron were each treated with 1% of active material of four chemically different types of antistatic agents in the conventional manner. Surface resistivity and volume resistivity measurements were made on these fabrics with the results shown in Table I. These results have also been compared with experience obtained from the use of the same antistatic agents in actual plant practice.

It is to be noted that all of the antistatic agents reduce both the surface and volume resistivity of all the fabrics, but that the relationship between the effects of an antistatic agent on one fiber does not hold true for another fiber. For example, the polyglycol and substantive anionic antistatic agent show the same resistivity for nylon, whereas, for Orlon, the resistivity of the polyglycol type is less than one-hundredth of the other. Another interesting observation is that the cationic antistatic agent shows a reduction in resistivity to 50%, whereas the substantive anionic shows the resistivity to have dropped to 10% of the untreated value. These results, which represent only a portion of the data already accumulated, show that, while antistatic agents may always show a reduction in resistivity, the amount of reduction is not necessarily a measure of the effectiveness of the antistatic agent. All of the data accumulated so far have shown that the comparison of the lowering of a fabric's resistivity by various antistatic agents cannot in any way give a true measure of the effectiveness or money value of these agents.

Resistivity data on untreated and treated fabrics are being accumulated and are being compared with other electrical data obtained from these same fabrics. These will be the subject of a future paper.

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New York Section——

DYEING AND FINISHING PIECE GOODS MADE FROM DYNEL-RAYON AND DYNEL-RAYON-ACETATE BLENDS*

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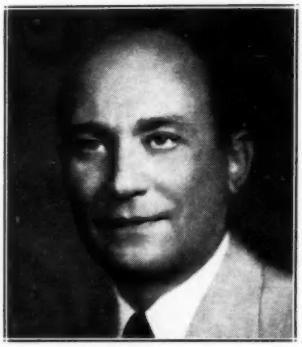
INTRODUCTION

THE successful use of the older fibers is the result of a vast amount of accumulated knowledge and experience in their handling. A thorough knowledge of the characteristics of the newer fibers is required if their best properties are to be put to work.

We shall discuss tonight the properties of Dynel that have to be considered in the dyeing and finishing of blends with rayon and with rayon and acetate.

To know these properties will make the difference between making a saleable cloth with excellent characteristics, or a piece of goods that looks blotchy, will change in ironing, or may be too stiff. In our publications we emphasize a few simple steps that have to be taken to master the possible difficulties, but we find that some people overlook our suggestions and then believe that the processing of Dynel requires measures that are hard to control. This is not so.



F C Rippner

Dynel is used in blends to improve hand, insulation, fabric stability, and beauty but primarily to gain crease retention.

The Dynel of piece goods containing also acetate and/or rayon is most often dyed with disperse (acetate) dyes, which also dye the acetate somewhat heavier. The Dynel may also be dyed with neutral-dyeing premetalized dyestuffs near the boil without special assistants. Several acid and milling dyes may be employed for light and medium shades on the Dynel; heavy shades with these types, however, are dyed in the presence of cuprous copper. The rayon of blends is colored with direct dyestuffs. When cuprous copper is necessary in dyeing certain heavy shades on the Dynel with acid-type dyes, four direct dyes that stand reduction are recommended for the rayon.

Since Dynel must be relustered by the action of heat either in a hot sodium sulfate bath or in air or steam, the ways necessary to carry out this action are explained. The advantages of the fact, for blends, that Dynel is thermoplastic are pointed out, and the importance of the proper relaxation of Dynel fabrics is emphasized.

DYNEL PROPERTIES AND PROCESSING

DYEABILITY———The first property of Dynel I want to emphasize is its dyeability. There is a misunderstanding on this matter even among people who have had frequent occasion to work with the fiber. They resort, more often than not, to unnecessary steps and the use of unnecessary agents. Actually, the dyeability of Dynel is extremely good, and several dyestuff groups can be applied. However, temperatures at, or close to, a boil give best yields.

Disperse (acetate) dyes are most universally used. They dye Dynel very well, almost without exception. A number of them have excellent fastness properties. No swelling agents are used with acetate dyes. They give good yields, and the question of temperature is not critical. Even navy and black shades can be dyed on open equipment. The yield improves greatly with higher temperatures. However, I would like to mention this: the dyehouse that has dyed the largest yardage of Dynel blends does not have a single piece of closed equipment on its premises.

A number of the newer premetalized dyes have good affinity for Dynel, and many of them show excellent fastness properties — fastness to washing at 170°F and high light fastness in the Fade-Ometer. A few brand names of the neutral-dyeing premetalized dyestuffs include Vialon, distributed by Nova Chemical Corporation; Nyalan, sold by Nyanza; Irgalan, of Geigy; Cibalan of Ciba; Lanasyn of Sandoz; Capracyl of DuPont; and Supralan of General Dyestuff Corp. Not all of these dyestuffs have the best properties, but a good selection can be made.

Several acid and milling dyes can be used on Dynel. They yield light to medium shades without the use of any agents, and deep shades with reduced copper.

LUSTER———The second property of Dynel of importance is its tendency to lose its luster in water at or near the boil. Delustered Dynel will show all shades, including its own natural color, much lighter than the lustrous fiber. The relation is somewhat like the relation of color yield on dull and bright viscose dyed in the same bath. There are three ways to restore luster. One is to heat the material to 240°F for 5-10 minutes. Another is to steam the fabric, as in decating at 40 to 60 lb per sq inch for 2 to 5 minutes. The third and least desirable way is to boil Dynel in a heavy solution of sodium sulfate for several minutes. While the heavy-salt-solution technique for relustering is normally used in the package and stock dyeing of all-Dynel, it is not practical for blended piece goods. However, it gives the dyer a simple method of relustering his handpatches properly. The normal procedure for blended piece goods is to take advantage of the heat-treatment in curing resins during the regular finishing procedure.

HEAT SENSITIVITY———The next property to be discussed is the reaction of Dynel to heat. It is more heat sensitive than many of the commercial fibers of today. At first glance this characteristic may appear to be a deficiency. However, it actually is advantageous for several purposes. We know that certain of the thermoplastic fibers impart crease-retentive properties in blends. But, as we also know, most of them will retain the set creases very stubbornly. They are very difficult to remove. With Dynel and good Dynel blends, however, wrinkles set in

* Presented at Kohler's Swiss Chalet, Rochelle Park, N J, on Nov 20, 1953. Submitted to the REPORTER by the Editor of Proceedings on Feb 25, 1954.

by improper handling or creases pressed in by a tailor, for that matter, can be removed easily by further pressing. This is a direct result of the fiber's higher heat sensitivity.

After dyeing, Dynel or Dynel-blend piece goods should be cooled slowly to avoid setting wrinkles. But if, by accident, the material shows wrinkles, they can be removed by boiling them over and cooling properly. No irreparable damage to the goods is caused.

Another important factor is the fact that Dynel shrinks when heated. We ourselves were at one time afraid to treat Dynel blends over 250°F. While this upper limit still stands for 100% Dynel fabrics, we learned that the normal curing temperatures for resins is not only permissible for Dynel blends but the heating of fabrics to resin-curing temperatures is the only proper way to handle them. Here is the reason: the heat treatment will, as above mentioned, cause the Dynel to shrink. It will shrink away from the surface and thus cause a change in appearance and shade. If the piece being treated is not under tension, it will also shrink in width and length and thus become dimensionally stable and stable to further shade change in pressing.

As just mentioned, the heat treatment will produce a change in appearance. This fact caused some dyers and finishers to throw up their hands and exclaim that Dynel cannot be processed. The reason was usually a piece of uneven fabric, shaded from side to center, uneven and blotchy. But a simple run through a dryer at 300°F, as if by magic, changed the same piece to one that was even all the way through.

May I give you a brief explanation of this occurrence? In most plants, for reasons of efficiency, the goods are kept in the dryer just about long enough for the entire cloth to be dried. Temperatures in some ovens vary, and one part of the cloth may be fully dried, as in a loop dryer, before it reaches two-thirds of the run. The Dynel in this part will have ample time to reluster and to shrink into the cloth and show the final appearance desired. Another part of the piece, usually the one at the lower end of the loop, will be just about dry as it leaves the oven. Neither is this part sufficiently relustered, nor is the Dynel shrunk into the fabric. This area, therefore, will show up much lighter. Now, as mentioned before, if one were to run this piece through the oven again, it will all level out, always presuming, of course, that the dyeing was level. Actually, the second run through the dryer isn't even necessary. The normal curing process for the resin applied to the fabric will take care of the leveling out. After a while, management and workers will get used to the fact that an occasional piece of Dynel blend may look blotchy after the first run through the dryer. I emphasize this, as we sometimes receive calls for help in cases where there is really nothing wrong.

SOME DETAILS OF PROCESSING

The processing of Dynel-blend piece goods is based entirely on the three properties mentioned. To explain further, I would like to give you now a step-by-step outline of the scouring, dyeing, and finishing of rayon-acetate-Dynel and rayon-Dynel blends.

I wish to emphasize that these procedures and suggestions are based on practical experience. They do work. Beautiful merchandise is being produced in flannels, gabardines, plain weaves, twills, and so on. Some of the steps may be a little unusual, but none of them require special equipment or undue attention.

SCOURING———Considering the thermoplastic properties of Dynel, the setting of creases in the scouring operation should be avoided if possible. The Tensitrol machine is well suited for this job. However, many other ways are feasible as long as the hot goods are not suddenly chilled. The characteristics of the Dynel-blend goods are similar, in this respect, to those of acetate and acetate-blend goods. The simplest and safest method of scouring is to do it right on the beck at around 160°F. Very little difference has been found in the general quality of goods between those processed this way and those that have gone through a boil-off machine.

DYEING———The dyeing of rayon-and-Dynel blends is really very simple and can be handled almost like the dyeing of rayon-and-acetate. Practically all acetate dyes have good affinity for Dynel and they will build up even into good black shades. However, proper selection of dyes is just as important as on any other fiber. No dyer would dye a piece of goods without having at least some idea of the fastness requirements, cost limits, etc. May I urge you to select your dyestuffs by consulting our manual or the manuals of several dyestuff manufacturers? Well-selected dyes will result in better-dyed goods at lower prices.

Light and medium shades can be achieved easily even at temperatures around 190°F. The amount of dispersing agent should be kept to a minimum. Some dispersing agents — especially some nonionics — coagulate with certain blue acetate dyes especially recommended for Dynel. In using these particular dyes, nonionic dispersing agents should be omitted entirely. The manufacturers of these dyes are well aware of this fact and will gladly advise you on the matter.

On the subject of samples I would like to reemphasize the importance of the proper relustering of handpatches. I will have more detailed suggestions on this matter later on.

The dyeing of rayon-acetate-Dynel blends is not much more complicated in most cases than the dyeing of rayon-Dynel blends. For light shades, acetate and direct dyes should be used exclusively. Acetate dyes dye Dynel to a lighter shade than acetate. During the finishing operation, the Dynel will, however, withdraw into the cloth sufficiently to become inconspicuous. Medium shades, too, are often handled in this way. When it comes to the heavier shades, neutral-dyeing premetalized dyes are of great help. These dyestuffs have a direct affinity for Dynel and can be added to the bath right along with direct and acetate dyes. With proper selection. excellent results can be achieved. The line comprises yellows, reds, browns and maroons. However, there is only one blue with sufficient affinity: Capracyl Blue G. This dye yields somewhat dull shades with medium lightfastness.

Thus, for the very best results in dyeing navy, dark-green, and black shades on these Dynel blends, there are two alternative ways open to achieve a good union. The first one is to use a swelling agent. These agents will help the acetate dyes dye the Dynel deep enough to give a good union. As dyehouses do have objections to the use of swelling agents, I would like to outline a very simple application of reduced copper to achieve good unions in the navy, dark green, and black shades. Proper selection of dyestuffs, again, cannot be overemphasized. The direct dyestuffs should be resistant to reduction. The acid dyes to be applied on the Dynel should be those that have especially good affinity for Dynel so that a minimum of reduced copper can be used. There are four direct dyestuffs that have been tested for resistance to reduction and that do an excellent job. They resist reduction not only by the reducing agent used to reduce the copper but also by prolonged boiling in closed becks; they level well, are light fast, and are not influenced excessively by resin application. They are the following or corresponding prototypes:

Solophenyl Blue 2 RL
Solophenyl Blue 6 GL
Solophenyl Orange TGL
Solophenyl Bordeaux 2 BL

The acid dyestuffs recommended are these:

Xylene Milling Blue GL and BL
Anthraquinone Blue SWF
Cloth Red G
Xylene Milling Yellow S
Neutral Orange GX

The dyeing proceeds as follows: The dyebath is made up at 100°F, and all the dyestuffs are added at once. A small amount of a good anionic dispersing agent should be used. High amounts of dispersing agents will retard the absorption of the dyestuffs on the Dynel. The bath should be brought to a boil in about 40 minutes, boiling is continued for about 20 minutes, whereupon the salt may be added. The salt has to be sodium sulfate, as common salt will interfere with the action of the reduced copper. Any shading of the viscose and acetate is preferably done at this stage. When the direct and acetate dyestuffs are reasonably exhausted, the reduced copper can be added. Add about 0.1% of sulfuric acid to the dyebath to give it a pH of about 6-5. However, no other acid should be used. To make up the reduced copper, 1% anhydrous copper sulfate should be dissolved in a small amount of warm water. The solution should be cooled by adding cold water. Four-tenths percent of zinc formaldehyde-sulfoxylate should be dissolved separately in lukewarm water. Then, the two solutions can be mixed and added slowly over a period of about 20 minutes to the dyebath. The addition of the reduced copper will drive the acid colors on the fiber without difficulty. Slow addition will insure level dyeing. I never have seen an uneven dyeing on Dynel caused by the addition of reduced copper.

A simple device in color matching is the use of "fliers" or "telltales". In a three-way blend it is often impossible to tell which of the fibers is off shade. The "telltales" will easily give the story. The hand patches should receive the same heat treatment as the goods will receive in the finishing department. This is necessary to reluster Dynel fully. If, however, proper heating equipment is not available, a boiling 20% solution of sodium sulfate will do the same thing. Just simply drop your all-Dynel hand patch into the boiling solution for about 5 minutes. This will reluster the Dynel. A very close match on the Dynel is really not necessary because the fiber is barely visible after the finishing and will hardly influence the final shade of the blend.

SLOW COOLING OF DYED DYNEL ——As mentioned beforehand, it is important to cool the goods slowly. A good recommendation is to shut off the steam, open the doors if working in closed equipment, let the goods run for ¼ of an hour, and then slowly add cold water. An overflow rinse should then be given. If you find creases in the finished goods, it was due to too fast cooling. As the damage is not permanent, the goods can be boiled over again in a heavy Glauber's-salt solution to avoid loss of dye, cooled slowly, and rinsed.

For the extraction the usual precautions are sufficient.

FINISHING———Now, to the finishing. The goods should be dried like most quality goods. This means that temperatures should be kept to a practical minimum. It will help to eliminate dyestuff migration. This applies to acetate-viscose goods as well as to fabrics containing Dynel. Light and medium shades can be put through the loop dryer. With darker shades, it is advisable to dry on a covered frame. Several dyehouses apply a temporary dye-fixing agent to overcome color migration and are then using loop dryers successfully for even the darkest shades.

Should the goods look blotchy, uneven or shaded at this stage, make sure you don't send them back to the dyehouse before checking these points. Take the worst piece and run it through the loop dryer at 300°F. In 99% of the cases, the goods will come out level. If so, apply the resin finish as usual and dry on the tenter frame-Palmer unit, if desired. One piece of equipment that sometimes causes difficulties in drying the resin is the covered tenter frame. This happens if the goods don't dry completely or evenly on the first run. Overheated clips and acetate dyes that are sensitive to resin may result in irreparable shading of edges.

For curing, run through the loop dryer at 300-320°F. From here, the goods may go on a frame again where they should be run while very slack. In order to achieve better dimensional stability of the cloth, we urge every finisher to run the goods absolutely relaxed during all drying and finishing operations. Light steaming on the frame is beneficial. However, do not try to remove all the creases in this operation. Rather, apply a semi-decating for several minutes. This will not only remove the creases, but it will increase dimensional stability and help overcome difficulties if the goods should be used for pleated skirts. Again, properly selected dyestuffs and semidecating will help to produce goods that will not change in the harsh treatment of pleating — assuring the makers of two-piece ensembles that the pleated skirts look exactly the same as the blouses.

I have tried here to point out how you can use your equipment most economically to handle blends of Dynel with rayon and the three-way blends with rayon and acetate. The techniques are those you use every day — all that is needed to obtain attractive merchandise of these blends is a recognition of the properties of Dynel. The fiber's dyeability, its luster and its response to heat can all help you obtain excellent results. It is most gratifying to see the goods now being turned out by many dyeing and finishing concerns. It was through their splendid co-operation, dyeing and finishing know-how and assistance that the fabrics produced at this stage are excellent in shade, have good stability and shrink-resistance, and very good fastness to washing, to sunlight and to pleating. This combination of fabric characteristics — which you determine — produces saleable merchandise with tangible economic advantages. I wish to take this opportunity to thank the dyestuff companies also for their very valuable contributions.

I will be glad to try to answer any questions you may have.

DISCUSSION

Question: Is it practical to dye Dynel-acetate-rayon blends by a two-bath procedure and thus to increase the range of dyes that can be used for the viscose in the presence of a reducing agent?

Answer: Only a few shades, such as navies, dark greens and blacks have to be dyed by the copper method, and the four dyestuffs named are sufficient to dye those shades.

Q: What is the optimum amount of Dynel recommended in fiber mixtures to raise the softening point? I understand that 240°F is the highest finishing temperature for an all-Dynel fabric. Can you go higher if you have 70% viscose in a Dynel blend?

A: We normally recommend no more than 35% of Dynel in blends. Three-way blends usually contain 25% Dynel, 25% acetate, and 50% viscose rayon.

I have here some samples containing 65 to 75% of viscose and 35 to 25% of Dynel, which should be treated at 300 to 325°F, a temperature considerably higher than the 240°F formerly mentioned.

Q: If Dynel gives dimensional stability to a blend, is resin finishing then necessary? What percentage of Dynel do you recommend for obtaining dimensional stability?

A: The major contribution of Dynel to a blend is crease retention; it does furnish some dimensional stability but not always enough. Furthermore, a resin finish stabilizes the rayon and also very importantly, gives the rayon wrinkle resistance. The crease retention resulting from the admixture of Dynel permits a pair of slacks, for example, to retain a crease even when the wearer is caught in a rain shower.

Q: Does Dynel impart any other properties?

A: Oh, yes. First of all, it gives a

pleasant hand that is very hard to duplicate by the addition of any other fibers. In many blends it gives good heat insulation and an appearance that is hard to imitate. Dynel also adds much to the strength and wear life of a fabric.

Q: How do you obtain a good light union (tone-in-tone) shade on a three-way blend?

A: For light shades, use disperse (acetate) and rayon dyes only. The acetate colors will dye the Dynel lighter than the acetate, but when the Dynel withdraws into the mixed fabric in finishing, the small lack of color on the Dynel will hardly be visible. Even gabardines can be handled that way, and nobody will be able to tell the difference.

Q: In the case of high-pile cloaking materials, what is the so-called electrifying process they speak of, which is being carried out by two or three concerns?

A: I do not know the details of the process, but I think it is something like moutonizing.

Q: With most acrylics there is the problem of static electricity. Do you run into that problem with Dynel?

A: Yes, we do. But in blends containing viscose or viscose and acetate, static electricity is almost zero. In the case of all-Dynel, static electricity is quite a problem, but antistatic agents help out.

Q: What about the fastness to washing of antistatic agents?

A: To date commercial antistatic agents are not claimed to stand more than a few washings.

Q: What are the pilling properties of Dynel?

A: Dynel fabrics pill much less than those of some competitive fibers. Suits are now being sold in Manhattan made of wool and Dynel. We guarantee to replace any garment that shows pilling; so far we have not had one suit returned.

Q: Do you recommend brushing material, such as a flannel, made from a Dynel blend?

A: Flannel goods are usually napped. There is no difficulty in napping Dynel blends at all.

Q: What swelling agent do you recommend for dyeing Dynel?

A: Well, there are a number of brand names, but I do know that several dyehouses use trichlorobenzene; at least one dyestuff firm recommends phenylmethylcarbinol.

Q: The shrinkage characteristics of Dynel fabrics have been mentioned. Do you allow for that in the construction of Dynel materials?

A: In the construction of Dynel blends or all-Dynel goods there definitely should be an allowance for shrinkage, which is somewhat higher than for viscose-acetate materials. An acetate-rayon-Dynel fabric shrinks about 12½% during full stabilization.

Q: Has any attempt been made to measure the heat retention of Dynel fabrics against the heat retention of fabrics containing no Dynel, or against those containing the same amount of wool?

A: Yes, many tests have been made. The advantage of Dynel over wool is that, after several washings, a Dynel fabric retains its insulating properties much better than one containing wool.

Q: Have you the data?

A: Not at hand, but we will be glad to furnish them.

Q: What is the average shrinkage of a Dynel decorative fabric? What do you do, for example, for a 52-inch all-Dynel curtain fabric?

A: The shrinkage of an all-Dynel materiel depends more upon construction than does the shrinkage of a part-Dynel material. The percentage must be determined for each construction; I cannot give a single figure.

The goods should be boiled at a full boil for an hour or so on an overhead reel with lots of water so that the goods are not dragged out of the bath and relatively little weight hangs on the upper part of the piece. In this way the fabric is not stretched and has a good chance for relaxation. The fabric should then be loop-dried at 250°F for about 15 to 20 minutes. Full relaxation will then be permitted and no more shrinkage will be left in it.

If you have a small piece, boil it in a beaker and count the picks and warp ends per inch both before and after boiling to get the shrinkage. As the shrinkage of any construction can only be approximately estimated ahead of time, the exact value for any Dynel fabric must be experimentally determined after weaving.

Membership Applications

March 12, 1954

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• PATENT DIGEST •

PAUL WENGRAF

COATING COTTON WITH VISCOSE———Steam and Wetting Pretreatment G, 2, 01

U S Pat 2,639,966
(Textuff, Ltd——Stanton——May 26, 1953)

The coating of woven or knitted fabrics with a viscose solution that will subsequently coagulate frequently has been advocated as standard procedure. One of the pertinent methods, described in Brit P 611,275 (see reference below), comprises a pretreatment of the cotton fabric with live steam to eliminate absorbed air so as to avoid formation of air bubbles or "pin holes" in the finished goods. According to the current invention pin holes nevertheless may occur due to the development of gases in the decomposition of the viscose coating. Moreover a tendency has been observed for *minute blisters* or protuberances to develop, due to little cotton fibrils to which the coagulating viscose may adhere.

Satisfactory results are reportedly attained by combining the steam pretreatment with a passage through a hot wetting-out solution. Preferably the viscose solution is permanently circulated through a filtering device, which will retain the fibrils detached from the fabric.

As illustrated in Figure 1, the fabric (1) runs through a steam chamber (2), then through the wetting solution (4); it is air-cooled before entering the viscose solution (6), leaving the container (5) by slot (7). After being tentered, the fabric is rinsed (8), tentered again and dried. Part of the liquid (6) is permanently withdrawn by pump (15) and circulated through screen filter (16) and cloth filter (17) before re-entering the container (5).

Dyeing of the fabric by adding direct dyes either to wetting solution (4) and/or to viscose solution (6) is within the scope of this patent.

Among the references cited by the Patent Office:

U S Pat 2,378,186 (Sylvania Ind Corp/1945): impregnating artificial silk material with alkaline dispersions of alkali-soluble, water-insoluble ethers of cellulose and coagulating this substance while maintaining the fabrics in a tensioned state.

Brit P 611,275 (Courtaulds/1948): pretreating the fabric with live steam before coating with regenerable cellulose solutions.

Brit P 280,262 (Brit Dyestuffs Corp/1927): improving penetration of various materials to be impregnated by a pretreatment with a wetting-out solution containing an alkylaromatic sulfonate.

AZODYEING CELLULOSE ACETATE———Coupling of Components Improved C, 4, 07

U S Pat 2,643,175

(Celanese Corp of Am——Salvin——June 23, 1953)

While the dyeing of cellulosic fabrics with developed naphthol-base azodyes is standard procedure, giving light- and crocking resistance, e g, in true blue shades, the application of this method on cellulose acetate invites trouble, especially in heavy blue colors. The results differ from those on cellulose in that plum and purple shades result, which lack wash-, light- and crockfastness properties.

Certain modifications were found to be necessary in order to avoid the undesirable formation in the diazotizing operation of nitroso naphthols, which produce off-shades and a lack of lightfastness. It has been observed that low acidity in a diazotizing bath containing organic acids (preferably acetic acid) rather than mineral acids is apt to reduce the formation of nitroso compounds. Moreover, an aftertreatment with a hot alkaline soap solution (75-90° C) improves penetration and promotes coupling. Alcohol, glycol or Cellosolve optionally may be added as swelling agents to the soap solution or applied in a separate aftertreating bath. The feature of this patent is the combination of low acid diazotation, developing and swelling.

Example: an all-cellulose acetate fabric was dyed with a mixture of Naphthol AS and Fast Blue BB base, diazotized in a bath containing sodium nitrite and acetic acid and soaped after rinsing for one hour at 85-90° C. The resultant shade was a deep-violet blue, having a 20-hour lightfastness (Standard AATCC). Developing for 1½ hours at 65-70° C gave a purple blue with only a 10-hour light fastness. Thus it can be seen that the temperature of the developing bath is important. Other examples call for aftertreatments with swelling agents.

Among the references cited by the Patent Office:

U S Pat 2,218,628 (Celanese Corp of Am/1940): coloring acetate of cellulose by running the filaments through a dyestuff solution containing mixed solvents and swelling agents, e g, ethylacetate, ethanol and water.

U S Pat 2,048,786 (Celanese Corp of Am/1936): dyeing acetate of cellulose by applying thereto (in the free state) dimethoxy anilide of oxynaphthoic acid and coupling it with a diazotized diamino diarylamine.

U S Pat 2,048,796 (Celanese Corp of Am/1936): impregnating acetate of cellulose with azodye components and adding a reducing sugar to the alkaline solution of the naphthol.

Brit P 470,333 (Brit Celanese/1937): improving fixation of dyes on cellulose acetate by passing the fabric for a short time through vapors saturated with organic solvents.

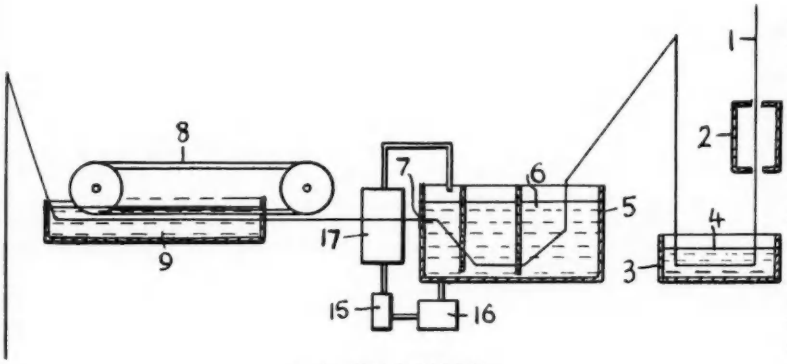


Fig 1—USP 2.639.966

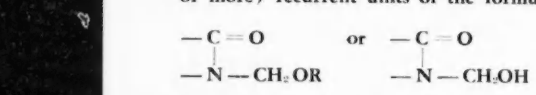
SHRINKPROOFING WOOL with Solutions of Water-Insoluble Polyamides A, 2, 04

U S Pat 2,644,773
(Montclair Research Co, Ellis Foster Co——Hammer, Rust——July 7, 1953)

The preamble to this patent enumerates certain disadvantages observed in standard shrinkproofing methods, for instance, the change of the normal hand of the wool material, fiber degradation, poor stability of the effect and of the substance deposited in the fibers, and so on.

A satisfactory shrinkproof wool is reportedly obtained by a treatment with organic solvent solutions of a water-insoluble polyamide, which can be applied in a rather wide range as long as the amount is insufficient to deteriorate the natural hand of wool, e g, 1-25% polyamide on weight of fiber (owf). Impregnation with the polyamide solution is followed by insolubilization by heat, generally by simply drying at temperatures not exceeding 100° C and preferably between 60-80° C, but optionally by vacuum drying. This aftertreatment avoids consequent overheating of the fibers or precipitation of the resin in a separate step.

The polyamides used are of the nylon or Perlon type and are generally characterized by an intrinsic viscosity of at least 0.4. Polyamides of this class are known to be soluble in strong acids and phenols. According to most of the examples, formic acid from 70-100% concentration, and preferably of the commercial concentration of 90%, is used. Since these strong solvents may cause some difficulty in handling, however, it is suggested that modified, alcohol-soluble polyamides be applied containing at least partly (10% or more) recurrent units of the formula



where "R" is the lower alkyl, provided that their intrinsic viscosity is within the above limits. Products of this type are prepared, e g, by treating a) the original polyamides with paraformaldehyde and b) by alkylating the OH groups thus formed.

Example: 10 p of a commercial polyhexamethylene adipamide are dissolved at 60-70° C in 90 p anhydrous formic acid. Wool is immersed in this solution and squeezed to a pick-up of 25%, then dried at 60-70° C, relaxed for 2 hours in a 1% soap solution and laundered for another 2 hours in a 0.2% soap solution at 65° C. The treated sample, when compared with an untreated specimen, shows a total shrinkage of 18% against 35.8% for the control sample and a substantial improvement in shrink-resistance after laundering.

Among the references cited by the Patent Office:

U S P 2,526,637 (Du Pont/1950) [cf *Am Dyestuff Reptr 40,* 198 (1951)]: using high-molecular-weight basic amino polymers reacted with formaldehyde for shrinkproofing wool.

U S P 2,443,450 (Du Pont/1948): coating with N-alkoxymethyl polyamides which have been obtained by reacting the polyamide with a mixture of formaldehyde and alcohol. U S P 2,412,993 (Du Pont/1946) discusses the method for preparing the N-alkoxymethyl polyamides.

U S P 2,430,860 (Du Pont/1947): preparing formaldehyde reaction products of linear polyamides in the presence of an oxygen-containing acid as catalyst.

U S P 2,406,958 (Du Pont/1946) [cf *Am Dyestuff Reptr 36,* 37 (1947)]: reacting wool with iodine dissolved in an inert liquid (styrene) to render it shrinkproof.

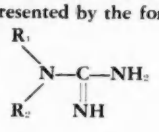
U S P 2,347,024 (Beer/1944): impregnating textile materials with mixtures of urea-formaldehyde resin precondensates and Zelane.

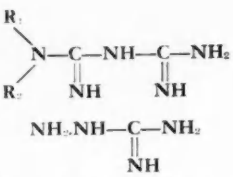
DYEING NYLON OR CELLULOSE WITH ICE COLORS———Guanidine Added C, 4, 07

U S Pat 2,649,353
(General Aniline & Film Corp——Streck——Aug 18, 1953)

A well known method of dyeing cellulose acetate or nylon with insoluble azo-dyes comprises impregnating the fibers with a mixture of the coupling components (naphthols) and the diazotable base in one bath and diazotizing in another to form the dye on the fibers in situ. The preamble to the current patent states quite generally that unsatisfactory, weak, and off-shade dyeings of poor fastness result with this dyeing method. This is especially true for nylon dyeings. A statement pointing out these facts is quoted (*Am Dyestuff Reptr* of Oct 17, 1944, p 748). These disadvantages are probably caused by the low affinity of the fibers to the naphthol salts and by the quick decomposition of the diazonium components before coupling has taken place.

It has been discovered that dyeings of excellent fastness may be produced on these fibers by impregnating with the aforementioned mixtures in the presence of a guanidine compound. It can be proved that the coupling component forms a salt (which has been isolated and investigated) with guanidine, aminoguanidine, or biguanide or derivatives thereof represented by the formulas:





(wherein R_1 and R_2 represent hydrogen, alkyl, aryl or aralkyl).

These salts are said to have more affinity for the fibers than the coupling components themselves or their alkali salts. They are further said to impart greater stability to the diazonium compound and they also have a buffering effect in forming the azo dyeings.

Ice-color bases and coupling components which give satisfactory dyeings with this process on nylon and cellulose acetate are enumerated; 42 examples referring to the application of these mixtures blended with guanidine compounds are presented. Guanidine compounds should be applied at a ratio of 1-5% of the fabric weight (owf). When salts such as guanidine carbonate are used, the guanidine base must be set free by a small amount of alkali. It is advisable to add a slight excess of the coupling component.

Example: nylon is treated with a mixture of Naphthol AS-E and Fast Blue BB base, guanidine carbonate, some caustic, a wetting agent (Igepon T) and Cellosolve. These ingredients are dissolved in warm water and used for dyeing at 60-65° C. Thereafter the goods are rinsed, diazotized in a fresh nitrite-acetic acid bath at 60-65° C and finished as usual. A bright greenish-blue shade results that is much brighter and stronger than that of a parallel sample dyed in the absence of guanidine carbonate.

Among the references cited by the Patent Office:

U S P 2,385,719 (Cyanamid/1945): amine salts of nitrated phenols (including guanidine derivatives) are recommended as insecticides.

U S P 2,304,821 (Dow Chemical Co/1942) describes additional compounds of diarylguanidine and arylbiguanide salts of dinitrophenols for use as insecticides and also in pigment production.

Brit P 502,861 (I G Farben Ind/1939) states in the preamble (p 1, lines 42-45) that the affinity of naphthols (AS series) is considerably improved by the addition of organic bases, e g, guanidine.

Ger P 486,696 (I G Farben Ind): increasing the affinity of cellulose acetate to basic dyes by adding hydrated cyclic bases (hexahydro methylaniline) to the dyebath.

French P 643,323 (1928) discusses the pretreatment of cellulose acetate before metal mordanting with a swelling agent, e g, an alcohol, an organic acid, thiocyanate and other swelling agents (among them the guanidines).

DYEING SYNTHETIC FIBERS with Vat Dyes C, 4, 07

U S Pat 2,646,339
(Celanese Corp of Am——Ward, Haggerty——July 21, 1953)

Numerous methods for vat dyeing synthetic fibers, i e, cellulose esters, polyamides, polyurethanes, vinyl- or acryl-polymers, and so on, have been suggested. Results, however, have not been entirely satisfactory because the resultant dyeings generally have not been as resistant to light or washing as on other textile materials dyed with the same brands, and also because the alkalinity of the baths has caused saponification or degradation of the fibers.

According to the present patent, an aqueous dyebath (100 parts by weight) which will permit dyeing of these materials with a wide range of vat dyes should contain, in addition to the dyestuff and the reducing agent, an alkali carbonate (0.1-1 p b w), an alkanolamine, such as monodi- or triethanolamine (0.5-10 p b w), and a water-miscible wetting agent, such as butylcarbinol, Butyl Cellosolve or diacetone alcohol (3-25 p b w). No degradation or saponification of the fibers occurs in this bath. Many suitable vat dyes are enumerated.

Example: 0.2 p b w Ponsol Jade Green 20% (C I #1101), 0.5 p sodium carbonate, 2.5 p diethanolamine, 4 p sodium hydrosulfite, 1 p tetrasodium pyrophosphate and 20% butyl carbitol are dissolved in 300 p water at 50° C. 10 p cellulose acetate staple fiber are immersed in this bath for 15 minutes, the temperature is raised to 80° C and maintained at this point for 45 minutes. After the fiber is rinsed, the leucovat is reoxidized with dichromate-acetic acid at 60° C and soaped. A uniform jade green is obtained. Level dyeings of slightly different shade are produced on nylon, polyurethane fibers, glycol terephthalate (Dacron) and acrylic or vinylic filaments.

Another example refers to the use of Carbanthrene Red BN 20% (C I #1162).

It should be noted that dyebaths of this type are not effective on cotton.

Among the references cited by the Patent Office:

U S P 2,543,994 (Du Pont/1951) [cf *Am Dyestuff Reptr 40,* 375 (1951)]: dyeing acrylonitrile fibers with indigoid or thioindigoid vat dyes, alkalis and aromatic amines in the presence of a reducing agent and reoxidizing the leuco-vat dye.

U S P 2,524,811 (Interchemical Corp/1950): dyeing plastic articles by swelling them with a water-immiscible solvent in which the insoluble dyes are dissolved in the presence of an alkylolamine reaction product of a sulfonated oil. Sodium phosphate and sodium nitrite are used as diluents.

U S P 2,524,072 (Celanese/1950) [cf *Am Dyestuff Reptr 40,* 148 (1951)]: dyeing cellulose acetate with vat dyes after subjecting the fibers to a swelling action of a water-miscible organic base combined with diacetone alcohol.

U S P 2,259,515 (Celanese/1941): dyeing cellulose acetate fibers with dyes containing acid groups that have no affinity for the fibers themselves. Dyeing is carried out in the presence of a mixture of glycerol derivatives (e g, glycerol chlorohydrin) and acetic acid.

Brit P 622,676 (Marchington-Sutton/1949): dyeing acetate of cellulose with vat dyes by vatting with a base weaker than caustic alkali, e g, triethanolamine, combined with an alcoholic swelling. [See the inventors' parallel U S P 2,622,958 digested in *Am Dyestuff Reptr 42,* 739 (1953)].

PAD DYEING OR PRINTING CELLULOSE ACETATE——Alginate Thickened Alcoholic Liquids C, 4, 07

U S Pat 2,647,035
(Celanese Corp of Am——Cramer, Jr, Fritz, Jr——July 28, 1953)

This patent refers to former methods of dyeing cellulose acetate from dilute alcoholic dyebaths [cf among others U S P 2,428,833-36, *Am Dyestuff Reptr 37,* 397 (1948); U S P 2,519,498, *ibid 40,* 106 (1951), and so on].

It is essential that any migration of dyestuff solution applied in padding should be eliminated as migration during the skying of the dyed fabrics causes a mottled appearance of the dyeings. For this reason it is necessary to use sufficiently viscous pastes in printing in order to maintain sharp outlines of the patterns.

Standard thickeners, such as gum arabic or tragacanth, cannot be added to these alcoholic dyebaths of a lower alcohol content up to 70-80%. It has been found, however, that *alginates of alkylamines* (diethylamine alginate, dibutylamine alginate, etc) are well suited for this purpose. The dyebaths or printing pastes reportedly are stable and smooth and have no tendency to foam. Dyestuffs of different classes, such as Wool Fast Blue GL, Azorubinol 3GP, basic and acetate dyes, may be applied. The addition of sodium sulfate, used in other dyeing procedures, must be avoided since it precipitates the alkylamine alginate.

Example: a dyebath is prepared from 5 p Amacel Pure Scarlet III dissolved in 306 p ethanol 95% and 125 p water, the liquid being thickened with 12.5 p dibutylamine alginate, which dissolves smoothly in the bath, forming a viscous paste of printing consistency. The printed fabric is dried, soaped at 40° C, rinsed in hot water and dried again. Sharp prints result.

References cited by the Patent Office:

U S P 2,514,410 (Celanese/1950) [cf *Am Dyestuff Reptr 40,* 64 (1951)]: printing cellulose acetate with pastes containing a chrome dye and a chrome mordant dissolved in dilute alcohol. Methyl celluiose is used as a thickener.

U S P 2,158,486 (Kelco Co/1939) proposes the use of triethanolamine salts of alginic acid for obtaining thickened, highly viscous solutions having good film-forming and adhesive properties.

PRINTING VAT DYES——Zirconium Salts Added D, 2, 01

U S Pat 2,650,152
(Am Cyanamid——Mecco——Aug 25, 1953)

This method is designed to overcome the poor stability of vat printing pastes in storage and especially after printing. Printed fabrics that cannot immediately be aged show a decrease in strength and lightfastness, particularly in a humid atmosphere.

This problem has been attacked by adding zirconium salts (oxychloride, tetrachloride, etc) to the printing paste. It has been observed that the anion of the salt is rather immaterial, the zirconium giving the effect desired. The theoretical reasons are not quite understood. Experimental results seem to indicate that at least part of the assistant reacts chemically and not merely catalytically. The amount of zirconium salts to be added to the paste is given as one-sixth to five times the weight of the dye.

Example (#10): a thioindigoid-type vat dye (C I # 1212, Cibared 3B or Calcosol Violet 6RD) is brought into uniform suspension and 1.5 parts of the real dye are pasted with 100 p starch-British gum thickener containing the necessary reducing and alkaline ingredients. Comparative samples printed with this paste and with other pastes wherein the thickener has been partly replaced by 0.25, 0.5, 1.0, 2.0 and 3 p zirconium oxychloride respectively, show increased improvement in color yield and brightness over zirconium-free prints. In another example (#4), two comparative samples of prints, one of them containing a zirconium salt, are stored in a desiccator for 18 hours at 80% humidity before aging. The print containing the zirconium salt is much stronger after developing than the control sample.

References cited by the Patent Office:

U S Pat 2,405,151 (Am Cyanamid/1946): using arylamine (aniline) salts of naphthoquinone- or anthraquinone mono- and disulfonic acids as vat dye printing assistants [cf *Am Dyestuff Reptr 36,* 37 (1947)].

U S Pat 1,945,484 (Gen Aniline Works/1934): printing thioindigoid dyes by adding to the paste salts, oxides or hydroxides of heavy metals. Cu, Fe, Sn or Zn salts are mentioned here, but not Zr salts.

ABSTRACTS

Solution-Dyed Staple

W W Bowman, *Textile Age* 18, 56-7, January, 1954.

Color through the spinneret in rayon and acetate is available today in both filament yarn and staple. Solution-dyed staple is the pigmentation of a fiber by the producer at the time it is spun. "Solution-dyed", or "dope-dyed", or "spun-dyed" are synonymous terms in common usage today for the same product. This addition of pigment in light or medium shades changes in no way the physical properties of the natural or white fiber, but merely incorporates color. This color is extremely fast to standard tests. In addition, this colored fiber is produced at a lower cost per pound than if vat dyed by the most efficient method.

Selection of the pigment or pigments used is a task in itself and requires the most careful testing of each. Just as various dyestuffs and methods of application vary in their fastness properties, so do pigments. Usually it is found that the fastness is weaker for the light shades and for the colors red and green than for the darker shades. The same applies to the pigments.

The author comments at length on the danger of contamination of white fibers when processed on machinery previously used for procesing spun-dyed fibers, and makes suggestions for avoiding this trouble.

He states that it is possible to blend the colored fibers with white fibers in various percentages and to obtain a multitude of shades or tints that are not possible without using two entirely different and usually much more costly fibers. Blends of white with black spun-dyed viscose fiber are capable of producing novel effects with but small percentages of the colored fiber. It is quite possible to obtain solid-appearing shades by blending some of the solution-dyed standard shades if it is not attempted to blend dark and light fibers or colors of widely different hue. A yellow can be blended with orange and a solid shade produced. In blending a dark blue and yellow, it is easy to obtain a heather or broken effect.

The advantages of solution-dyed fibers are enumerated as greater economy, superior fastness, more level dyeing, better penetration, and improved mechanical condition of the yarn (by elimination of the dyeing operation after spinning). The fastness properties are said to be as good as, and in some cases better than, those of comparable shades dyed with vat colors.

"Coloray" is Courtaulds' viscose rayon staple, and it is being offered in 13 colors at the present time with the prospect of several additional colors in the near future. Courtaulds have experimented with this process since 1938.—WHC

Some Notes on Wool Bleaching

Textile Technologist, *Textile Recorder* 71, #848, 159-60; #849, 81-2, November and December, 1953.

The author discusses first the practical aspects of peroxide bleaching, and stresses the importance of controlling the pH value of the bleaching bath. He recommends that this should never be allowed to rise above 9 because of the risk of alkali damage to the fiber. Residual bleach liquors, however, should be stabilized at a pH value of 3 or 4 with sulfuric acid, and again made alkaline when required for use.

For hosiery, the author states, the hydrogen peroxide bleach has largely replaced the sulfur process. Considerable quantities of wool are, however, still bleached by sulfur dioxide, the simplest method of carrying out this process being to expose the material in a wet state to the action of sulfur dioxide gas produced by the burning of sulfur.

The presence of water appears to be essential for the bleaching action, for if completely dry wool is exposed to dry sulfur dioxide, it acquires a yellow color and a great amount of SO_2 is absorbed. If, however, the wool is wet, a full bleaching action takes place.

Although details vary in different cases, the "stoving" or bleaching process is usually carried out by festooning the goods in a damp state from a series of parallel poles, or by some similar arrangement, in a wooden or brick structure. Crushed sulfur, piled in a heap around air nozzles, is burned in a hooded pot, a plentiful supply of air being maintained by a small fan.

An advantage of stoving over peroxide bleaching for wool hosiery goods is stated to be the production of a peculiar sheen which cannot otherwise be obtained. Nevertheless, the stoving process has several disadvantages, chiefly a) partial reoxidation, which causes a return of the natural yellowish color of the wool fiber on exposure during storage, and b) the partial destruction of dyes on subsequently colored yarns by the reducing action of residual sulfur. It has also been noted that stoved wool is noticeably slower in dyeing than is untreated wool.—WHC

Bleaching Nylon Fabrics: Use of Acid Solutions of Sodium Chlorite

Anon (British Nylon Spinners Ltd), *British Rayon & Silk J* 30, 64, December, 1953.

Occasions arise during the processing of nylon textiles when it is necessary to remove discoloration by chemical means. For example, hot-air setting sometimes causes slight yellowing of nylon fabrics, and this discoloration has to be removed. It may also be necessary to clear traces of identification tints from nylon, and also to strip dyed fabrics preparatory to redyeing. The use of acid solutions of sodium chlorite has been found effective for these bleaching operations.

Nylon textiles can be bleached satisfactorily by using the following conditions:

Concentration: sodium chlorite (80% conc), up to 3 lbs per 100 gallons of water
pH of solution (adjusted with acetic or formic acid): 3 to 5

Liquor-to-goods ratio: from 30:1 to 40:1
Processing time-temperature: 30 min at 85° C.

To stabilize the pH, additions may be made to the bleaching bath of tetrasodium pyrophosphate (½ to 1 lb per 100 gals), and a small proportion of a synthetic detergent stable under acid conditions may be added to assist in the bleaching action. The bleaching process is followed by neutralizing the fabric in soda ash solution, and finally by thorough rinsing in water.

Tenacity tests carried out on nylon yarn bleached in the laboratory for 30 minutes at 85° C indicated that under these conditions the yarn is unimpaired.

As acid solutions of sodium chlorite attack metal vessels, even including some types of stainless steel, the manufacturers recommend the use of either earthenware or wooden containers.

It is stated that the evolution of free chlorine dioxide, which is objectionable to personnel, can be avoided by the addition of 1 pint of 100-vol hydrogen peroxide per 100 gallons of bleaching solution.

Precautions should be taken to keep the sodium chlorite dry during storage and away from easily combustible materials, in order to avoid possible spontaneous combustion.

The usefulness of sodium chlorite for bleaching mixture fabrics containing nylon so far has not been ascertained, it is said.—WHC

Fluorescent Whitening Agents

J A Somers, *Brit Rayon & Silk J* 30, 62-3, December, 1953.

The commercial development of fluorescent whitening agents resulted from the discovery by Krais in 1929 that a fully bleached appearance could be given to half-bleached material by impregnating it with a solution of aesculin (6, 7-dioxycoumarin), which is present in horse chestnut bark. The lack of fastness to washing and light makes this unsuitable for commercial use by textile bleachers.

Real progress in the discovery of satisfactory fluorescent whitening agents came in 1934, when the possibilities of stilbene derivatives were investigated, for these could have a strong affinity for cellulose fibers, with fastness to light and washing, and a bluish fluorescence in ultraviolet light. Most of the commercial fluorescent whitening agents now available are derivatives of stilbene, and in particular, of 4,4'-diaminostilbene-2,2'-disulfonic acid.

In addition to substantivity for textile fibers and the necessary fastness to light and washing, other desirable properties in a fluorescent whitening agent are: good solubility in water in presence of electrolytes, acids and alkalis, high chemical stability, and no adverse effect on the fastness of dyes.

The author describes a number of stilbene compounds, substitution products of the one mentioned above, which have improved properties (e g, increased stability to active chlorine). He notes that it is not always essential or even desirable that a fluorescent whitening agent should have a pronounced affinity for textile fibers. When these agents are incorporated in washing powders to be used in laundries and in the home, it is undesirable that, with each wash, the whitening agent present in the detergent liquor should be absorbed by the textile material so strongly that there should be built up a high concentration of the agent. In such cases it is better that the substance left in the fabric or garment from a previous wash should wash out and be replaced by a fresh amount.

For laundry purposes, whitening agents are now being marketed which are based on coumarin products similar to the one first used by Krais.—WHC

Use of Silicones in the Textile Industry

D C R Miller, *Canadian Textile J* 70, #26, 72-5; 71, #1, 61-5, December 18, 1953 and January 15, 1954.

The purpose of this article is to examine in some detail those applications of the versatile silicone family of most interest in the textile field; first, from the standpoint of aids in textile manufacturing operations, and second, direct application to natural or synthetic-fiber fabrics.

Silicones can be produced in the form of volatile or viscous fluids, soft or hard resins, and gums or rubbers of many different types. The silicone fluids have a molecular structure of alternate silicon and oxygen atoms having one or more hydrocarbon radicals attached to the silicon. The length of the silicon-oxygen-silicon chain, the amount of cross-linking, and the particular combination of organic groups employed, cause the variation in the physical properties of the resulting product. The commercial silicones most used today are methyl, phenyl, or methylphenyl silicones, although as textile finishes the methyl hydrogen polymers seem to be of particular value.

Durability to weathering, resistance to heat and cold, and water repellency are the basic properties of all the present commercial silicones. These and the property of incompatibility with organic materials underlie all the textile uses for silicones.

Silicone greases are used as high-temperature lubricants (e g, for tenter frames, drying cans, etc). Silicone antifoam agents are effective wherever foaming is a problem. Silicone electrical insulating resins and varnishes, because of their heat-stability and moisture resistance, are valuable insulators for electric motors. Silicone masonry water-repellents preserve mortar joints and prevent efflorescence.

It was early apparent that their properties of durability and water repellence made silicones ideal for textile finishing if some means could be found of curing and bonding them to the fibers so as to resist laundering and drycleaning without necessitating temperatures which would damage or degrade organic fibers and dyestuffs. This problem has been solved by developing a series of linear methyl silicone polymers having some hydrogen-silicone bonds. Since this bond is labile, the hydrogen is readily removed with moderate heat, causing the resin to cross-link and become insoluble. Some of the silocone valences thus freed probably also bond to any free-surface valences on the textile fiber to provide a chemical surface bond.

Stable silicone emulsions which require only diluting with water and mixing with a catalyst are now available from several different sources. More recent improvements include a variation in the polymer itself to control the degree of cross-linking during the cure and thus control the "hand" of the treated fabric. These emulsions will impart to synthetic fabrics a soft, full hand or a hand having a crispness or resiliency equal to that of the finest worsteds, while at the same time producing a highly water-repellent finish, which does not alter the appearance of the fabric and is durably resistant to laundering and drycleaning. Furthermore, these emulsions are compatible with commonly used crease-resistant resins and permit simultaneous application of both finishes.

The author gives details of the application of the silicone resins to fabrics and the proper way to cure them. He states that in addition to water-repellency, properly silicone-treated fabrics have increased abrasion resistance, greater tear strength, reduced mark-off, improved sewability, and improved wrinkle resistance.

A silicone rubber, Silastic 131, which is used for coatings, is described. It is said to have extraordinary resistance to outdoor weathering, flexibility at extremely low temperatures, any desired degree of waterproofness, and ususually high rate of moisture-vapor transmission.—WHC

New Unshrinkable Wool Finish. Australian Process Uses Nylon.

A J Hall, *Textile Mercury & Argus* 129, 561-3, September 25, 1953

A new finishing process for reducing the felting power of wool, and capable of conferring substantial resistance to washing shrinkage in most types of wool materials, has recently received considerable publicity. It consists of impregnating the wool material with a modified nylon in alcohol solution, then wholly or partly drying it, and regenerating the nylon by a heat treatment or by simple treatment with a dilute aqueous acid liquor. The object is to deposit on the wool fiber surfaces a very thin continuous film (about 2 percent on the weight of the wool) of regenerated nylon.

As is well known, nylon is a generic term which covers a large number of polyamides closely related in chemical constitution. The nylon which is now so widely known and much used results from the reaction of adipic acid with hexamethylene diamine, and it is this nylon which is favored for use in the new unshrinkable wool finish. However, this nylon is not used as such, but is first converted into its N-methoxymethyl derivative by treatment with formaldehyde and methyl alcohol. This conversion process is described by the author.

The wool material is immersed in a solution of the converted nylon in ethyl alcohol at 30° C, extracted, and dried. The nylon is then regenerated by a treatment with dilute hydrochloric acid at 25° C, or by heating the material at about 120° C. On drying, it is stated that the wool is found to be substantially unshrinkable, and the abrasion resistance is increased. The effect of the treatment is said to be not readily removed by washing.—WHC

The Correlation of Dyeing Methods and Serviceability of Wool

Link, *Textil-Rundschau*, 279-301 (June, 1953)

The author, in an extensive study, has investigated the chemical and physical influences apt to deteriorate wool in wearing. Supplementing earlier research, which concentrated on the effect of weather on army cloth, the present work aims at re-examining and completing results reported in current literature, using chemical and physical methods. In the past, dyestuff groups such as chrome, vat, and others, have been included in these studies.

A discussion of the chemical and morphological constitution of the wool molecule, pointing to its amphoteric character, and a full description of the main dyeing operations using dyes of the acid, chrome, chromium complex, and vat (preferably indigoid) types and sulfuric acid esters of leuco vat dyes is offered. The mechanism of the dyeing process is explained and a valuable discussion of the theory of chrome-complex dyeing is presented. Another important section is devoted to the problem of damage to wool by chromium compounds.

Contradictory opinions are reported, particularly in the comparison of the effects of chrome dyeing and vat dyeing. Apparently both methods employ insulation as a protection against damage, and both have about the same effect on the ultimate tensile strength of the treated fibers.

Since no universal method exists for testing wool damage by different factors, such as the dyeing process, light rays, scouring, etc, the author presents a method for analytical determination based on the comparison of specimens with samples damaged to a well-defined amount by known causes. Methods employed in the study of damage include microscopic investigation, reaction with ammoniacal silver solutions, examination of dyeing behavior, micro pH determination, etc.

The practical conclusions drawn in the experimental part, especially in regard to the influence of dye fastness on resistance to wearing, are of special interest.—(97 references; in German)—PW

Brightening Textiles with Fluorescent Compounds

Landolt, *Textil-Rundschau*, 337-45 (July, 1953)

Bleached goods which show a yellowish tinge have been customarily aftertreated with dispersions of blue dyestuffs. This results in a slightly gray shade, caused by reduced light reflection.

Modern brightening methods are based on a purely optical effect brought about by activating the molecules of the substratum. This phenomenon is explained theoretically by the transformation of invisible U-light waves into visible light of higher wave length. This process, which depends primarily on the light source, is graphically illustrated.

Light from ordinary bulbs contains negligible amounts of UV rays only and will consequently not produce the fluorescent effect when directed on fluorescent compounds. Brightening in daylight insulation will differ according to the hour of the day and to weather conditions. Fluorescent dyes (Rhodamine, Eosine, etc) are well known but substantial work was required to develop practical brightening, i e, colorless agents. A great series of appropriate brightening agents has been advocated but it seems advisable to set up a standard white, produced in the presence of fluorescent substances, to evaluate the comparative effect of commercial brightening agents.

The author discusses different types of fluorescent whiteners (the Uvitex products) that are suitable for use on nylon, animal and vegetable fibers and blends; soaps and detergents; etc, and presents data on affinities and fastnesses of the individual products.

In order to evaluate the fluorescent effect, it was necessary to develop light rays which are similar to average daylight (400-700 mμ) and which contain a constant quantity of UV rays. A photochemical method developed by Pinte and Rochas (cf *Bull Inst Textile France*) comprises filtering the rays (composed of daylight and UV components) in different spectral regions and comparing the figures obtained with those of a white reflected light of known standard composition.

Drawings of equipment and curves referring to results obtained by increased concentrations and to washfastness of the obtained specimens are included.—(34 references; in German)—PW

Salt Formation and Air Stability of Naphthol AS Products

Loewenfeld, *Melliand Textilber*, 324-7 (April, 1953)

The question as to whether Naphthol AS prepares dissolved in alkalies are present in the form of mono- or dibasic salts has not yet been answered definitely. Basing his assumptions on titration curves, the author suggests that three types can be present:

a) monobasic products, such as Naphthol AS-B, which become dibasic in the presence of formaldehyde by forming a methylol derivative [cf Neber, *Z Angew Chemie*, 411 (1937)]; in this case formaldehyde improves the air stability of the solution.

b) those products, such as Naphthol AS-OL, having dibasic character in themselves; these are not substantially improved in this respect by formaldehyde.

c) those products, such as Naphthol AS-LB, which have monobasic character in the presence *or* absence of formaldehyde.

The fact that products of group (c) behave as monobasic compounds in aqueous solutions, while the presence of methanol produces disodium salts, explains why alcohols are added to naphthol solutions by transformation of keto types into enol types. It is concluded that transformation of mononaphtholates into formaldehyde condensates in certain cases produces disodium salts giving higher air stability than the original monosodium salts.—(In German)—PW

New Method of Triboelectricity

J Am Medical Assoc 154, 163, January 9, 1954

In the "Foreign Letters" section of this publication a communication from France points out that triboelectricity (frictional electricity), the first form of electricity used in electrotherapy, has been previously produced by static machines. Professor Darmois reported on April 8 to the Academy of Sciences that this same triboelectricity may be obtained from polyvinyl chloride fibers submitted to a slight friction. This negatively charged static electricity produces an electric field of a size only limited by the formation of the spark; in the air it corresponds to a value of nearly 30,000 volts per centimeter of height. Dr Marc Iselin, chief surgeon of the Nanterre Hospital and a member of the National Academy of Surgery, thought that triboelectricity could be used in therapeutics by the simple application of bandages or underclothes made of polyvinyl chloride fibers without the use of any generator. For a year and a half F Deniker has been using this method at the Nanterre Hospital on 75 patients by general or local applications. It is reported that varying degrees of success were obtained in treating a wide number of afflictions; perhaps the analgesic effect of such bandages was most successful in the treatment of luxations, fractures, sprains, and trophic disorders. In his report of July 3 to the Medical Society of the Paris Hospitals, Deniker stressed, among other things, that the therapeutic efficacy of polyvinyl chloride was checked in 10 patients, during the course of the treatment, by substituting underclothing of regenerated cellulose. This was followed by a reappearance of pain and functional impairment in eight patients, and the other two reported continued alleviation of pain. The mode of action is not understood.

Book Reviews

(Concluded from page 192)

DETERGENCY EVALUATION AND TESTING

J C Harris
Interscience Publishers, Inc
250 Fifth Avenue
New York 1, N Y
210 pages
$3.75; 1954

This little book is another of the Interscience Manuals intended to describe laboratory methods and techniques in specific fields. No information that is not available elsewhere is supplied in this case but it is collected in this manual for quick reference. Procedures given are the author's selection and are not, in the reviewer's estimation, always the best or most accurate; however, they all are usable and certainly are given in sufficient detail even for laboratory technicians in other fields. The author is commendably careful to point out the dangers of applying data obtained by general detergency tests to specific cases.—JHS

TEXTILE FIBERS, YARNS AND FABRICS

Ernest R Kaswell
Reinhold Publishing Corp
330 West 42nd Street
New York 36, N Y
xvi + 552 pp
$11.00; 1953

The author has performed a notable service to all phases of textiles in the production of this book: "book" in this case is perhaps using the word too loosely —as Mr Kaswell in his preface states . . . "The book is actually an annotated and interpretive bibliography describing and discussing the research results of some 400 investigators." With such a statement this reviewer is in full agreement. The text therefore has some of the good features of a bibliography, for example, information is concentrated and easily accessible. Unfortunately, to the mind of this reviewer at least, such a text also contains some of the ill features of the bibliography, in particular, too much material must of necessity be included and the result often is a lack of clear-cut and decisive information. This undoubtedly is more the fault of the present state of "textile science" rather than that of the author.

The book is divided into two sections; Part A discusses the inherent properties of fibers, while Part B discusses geometric factors. In Part A such topics as Tensile Properties, Fiber Stresses and Deformations; Friction, Thermal Properties, Microbiological Properties, and the Dyeing Characteristics of Fibers are covered. In Part B such topics as Yarn and Fabric Geometry, Air Permeability, Water Repellency, Abrasion and Wear Resistance, and Fabric Hand are covered. Much information as to the present status of these factors is offered.

A thorough perusal of this text indicates that an understanding of textile materials, from a fundamental viewpoint, is incomplete and beset with perplexing problems. This of course has been known for a long time to the textile scientist, but not perhaps to the more practical worker. And to such a person a reading of this text will be of great value, both for the information it contains, and for that which it lacks!—VL

ORGANIC ANALYSIS, Volume I

Editorial Board: John Mitchell, Jr, I M Kolthoff, E S Proskauer, A Weissberger
Interscience Publishers, Inc
250 Fifth Avenue
New York 1, N Y
$8.50; 1953

This book is the first volume of a series on organic analysis, which the editors plan to continue as an annual publication. It is directed mainly at the practicing analytic and organic chemist and attempts to give a critical evaluation of many of the procedures of quantitative organic analysis currently in use. The present volume emphasizes functional group analysis and contains the following chapters, each written by experts in the respective fields: 1) Determination of Hydroxyl Groups, 2) Determination of Alkoxyl Groups, 3) Determination of the Alpha-Epoxy Group, 4) Organometallic Compounds for the Determination of Active Hydrogen, 5) Diazomethane for the Determination of Active Hydrogen, 6) Determination of Carbonyl Compounds, 7) Determination of Acetals, 8) Determination of Organic Sulfur Groups, and 9) Spectroscopic Functional Group Analysis in the Petroleum Industry. Except for the last chapter, emphasis is generally placed on chemical methods of analysis. Each chapter contains a large number of references to the original literature.

By its very nature, this book appears to be of great value to the chemist in the textile and allied industries. As specific examples of its applicability, the determination of free hydroxyl groups in cellulose derivatives (pg 32); determination of alkoxyl groups in cellulose ethers (pg 94) determination of organic sulfur groups in cellulose xanthate (pg 369), sulfonic acids and alkyl sulfates (pg 381) may be mentioned among others. It is hoped that the publication of subsequent volumes will not be unduly delayed.—JHR

ASTM STANDARDS ON SOAPS AND OTHER DETERGENTS (With Related Information)

ASTM Committee D-12
American Society for Testing Materials
1916 Race Street
Philadelphia 3, Pa
157 pages (heavy paper cover)
$2.25; March, 1953

This publication contains all of the ASTM standards and tentatives pertaining to soaps and other detergents in convenient form for industrial use. It is the result of the intensive research work carried out by ASTM Committee D-12 since 1936 involving round-robin tests, use of different apparatus, and related investigations in order to perfect standards satisfactory for testing and analyzing these products.

The book includes seventeen specifications for many types of soap and soap products, nine specifications for various special detergents, and nine methods of analysis covering soaps and other detergents. Definitions of terms applying to these materials are also given.

Additionally, the book contains the following appendices:

1) Proposed Methods of Test for Surface and Interfacial Tension of Solutions of Surface-Active Agents
2) Proposed Method of Tests for Rinsing Properties of Metal Cleaners.
3) Proposed Method of Test for Buffering Action of Metal Cleaners
4) Proposed Method of Total Immersion Corrosion Test for Soak Tank Metal Cleaners.—EPJ

TOXIC SOLVENTS

St Martin's Press
103 Park Avenue
New York 17, N Y
$4.00; Nov 20, 1953

This book commences with a well-written and interesting chapter on the characteristics of organic solvents. Some of the topics discussed in this chapter are: solvent capacity, solvent mixtures, boiling point and range, vapor pressure, evaporation rate, and flash points. The interesting fact, that the evaporation rate of a solvent is not in direct proportion to its boiling point but depends on many other factors, is brought out.

The next chapter is devoted to physiological effects to the nervous system, gastro-intestinal system, lungs, kidneys, and the blood.

The rest of the book deals with the individual solvents; their properties, industrial uses, toxicity, acute poisoning and treatment, chronic poisoning and treatment, and maximum allowable concentration are discussed.

This book should appeal especially to the chemist and to all those who in some way come in contact with solvents. Persons in the textile industry will find this book of definite value.—HB